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     (FILE 'HOME' ENTERED AT 15:21:35 ON 14 MAY 2006)

     FILE 'CAPLUS, MEDLINE' ENTERED AT 15:22:14 ON 14 MAY 2006
L1             0 S ?ACYL? GLUCO? (P) MIXED ANHYDRIDE?
L2            59 S GLUCO? (P) MIXED ANHYDRIDE?
L3             0 S L2 AND HALOFORMATE?
L4             0 S L2 AND HALOFOORM?
L5             0 S L2 AND HALOFORM?
L6             4 S L2 AND CHLOROFORMATE?
L7             0 S L2 AND BROMOFORMATE?
L8             0 S L2 AND IODOFORMATE?
L9             1 S L2 AND ALKYL ?FORMATE?
L10           18 S L2 AND ACYL?
L11           21 S L2 AND ACETYL?
L12           20 S ESTER? (P) CARBONIC ACID (P) GLUCOSE
L13            1 S HALOFORMATE (P) GLUCOSE
L14            1 S MIXED ANHYDRIDE (P) GLUCOSE (P) ?FORMATE?
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L6 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1992:592210 CAPLUS
DOCUMENT NUMBER: 117:192210
TITLE: Syntheses of N-acyl and N-alkoxycarbonyl derivatives of 2-[(alkoxycarbonyl)amino]-2-deoxy-D-glucose.
AUTHOR(S): Lafont, Dominique; Boullanger, Paul
CORPORATE SOURCE: ESCIL, Univ. Lyon I, Villeurbanne, 69622, Fr.
SOURCE: Journal of Carbohydrate Chemistry (1992), 11(5), 567-86
CODEN: JCACDM; ISSN: 0732-8303
DOCUMENT TYPE: Journal
LANGUAGE: English
AB N-Acyl and N-alkoxycarbonyl derivs. of 1,3,4,6-tetra-O-acetyl-2-alkoxycarbonylamino-2-deoxy-β-D- **glucopyranose** were synthesized using **mixed anhydrides** and sym. or unsym. pyrocarbonates. These derivs. were used as donors in 1,2-trans-glycosylation reactions promoted by Lewis acids. Besides the expected β-D-glycosides, cyclization and rearrangement side-products were often encountered in such glycosylations.

L6 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1991:470186 CAPLUS
DOCUMENT NUMBER: 115:70186
TITLE: Polyol polyester preparation in aqueous media using mixed anhydrides and use of the polyesters as fat substitutes in food and in cosmetics
INVENTOR(S): Lalezari, Iraj
PATENT ASSIGNEE(S): USA
SOURCE: PCT Int. Appl., 19 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9101322	A1	19910207	WO 1990-US4029	19900718
W: AU, CA, JP				
RW: AT, BE, CH, DE, DK, ES, FR, GB, IT, LU, NL, SE				
US 5498708	A	19960312	US 1989-381208	19890718
AU 9061881	A1	19910222	AU 1990-61881	19900718
PRIORITY APPLN. INFO.:			US 1989-381208 A	19890718
			WO 1990-US4029 A	19900718

AB Partially or completely esterified polyols, e.g. sugars, for use as dietary fat substitutes or in cosmetics are prepared in an aqueous medium by the reaction of the polyol with a mixed anhydride of a carboxylic acid and an alkyl **chloroformate**. A mixed anhydride was prepared from myristic acid (0.02 mol) and Et **chloroformate** in the presence of triethylamine (over an ice bath). To this was added sucrose (0.005 mol) and an oil formed. After extraction with petroleum ether and drying, a sucrose myristate was recovered (yield 97.4% based on sucrose tetramyristate).

L6 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1977:547477 CAPLUS
DOCUMENT NUMBER: 87:147477
TITLE: The preparation of carbohydrate-protein conjugates: cyanuric trichloride coupling of 2-aminoethyl glycosides, and mixed-anhydride coupling of 8-carboxyoctyl glycosides to bovine serum albumin
AUTHOR(S): King, Russell R.; Cooper, Fred P.; Bishop, Claude T.
CORPORATE SOURCE: Div. Biol. Sci., Natl. Res. Counc. Canada, Ottawa, ON,

Can.
SOURCE: Carbohydrate Research (1977), 55, 83-93
CODEN: CRBRAT; ISSN: 0008-6215
DOCUMENT TYPE: Journal
LANGUAGE: English

AB Preparation of the following glycosides is described: 2-aminoethyl β-D-glycosides of (A) 2-acetamido-3,4,6-tri-O-acetyl-2-deoxy-D-**glucopyranose**, (B) 2-acetamido-4-O-(2-acetamido-3,4,6-tri-O-acetyl-2-deoxy-β-D- **glucopyranosyl**)-3,6-di-O-acetyl-2-deoxy-β-D-**glucopyranose** (N,N'-diacetylchitobiose pentaacetate), (C) 4-O-(2,3,4,6-tetra-O-acetyl-β-D- **glucopyranosyl**)-2,3,6-tri-O-acetyl-β-D- **glucopyranose** (cellobiose heptaacetate); 8-carboxyoctyl glycosides of (D) cellobiose, and (E) N,N'-diacetylchitobiose. Conjugates were prepared from (A), (B), and (C) by coupling to bovine serum albumin by cyanuric trichloride and subsequent deacetylation; (D) and (E) were coupled to bovine serum albumin by the **mixed anhydride** reaction. Conjugates (A) and (B) were insol.; conjugates (C), (D), and (E) functioned as artificial antigens and gave rise to precipitating antibodies in rabbits. Specificities of the antisera were determined by inhibition studies.

L6 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1976:169742 CAPLUS
DOCUMENT NUMBER: 84:169742
TITLE: Lactam compounds and conjugates
INVENTOR(S): Bolz, Gunner; Leute, Richard K.; Soffer, Michael J.; Singh, Prithipal
PATENT ASSIGNEE(S): Syva Co., USA
SOURCE: Ger. Offen., 59 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2521523	A1	19760115	DE 1975-2521523	19750514
FR 2276317	A1	19760123	FR 1975-12665	19750423
FR 2276317	B1	19790427		
CA 1037475	A1	19780829	CA 1975-225944	19750430
JP 51001632	A2	19760108	JP 1975-61424	19750522
JP 63008427	B4	19880223		

PRIORITY APPLN. INFO.: US 1974-484026 A 19740628

AB Modified drug compds. were prepared which had a lactam function and were disubstituted on the C atom alpha to the carbonyl group. These compds. (hapten acids) were then conjugated to proteins, enzymes, or free radical mols. for use in immunoanal. For example, an aqueous suspension of 10.0 g Na diphenylhydantoin [630-93-3] was treated with an aqueous solution of 10.0 g chloroacetic acid and 10.0 g $NaHCO_3$ to give 2.0 g N3-(carboxymethyl)diphenylhydantoin (I) [741-28-6]. A solution of 124 mg I and 40 μl Et_3N in 4 ml anhydrous DMF was treated with 72 μl carbityl **chloroformate** at 0°, and the reaction mixture was added to 160 mg bovine serum albumin in 20 ml H_2O to give a conjugate with a hapten number of 51. **Mixed anhydrides** of hapten carboxylic acids with carbityl carbonate were prepared by treating a solution of 0.055 mmole hapten acid and 6.95 μl Et_3N in 0.5 ml DMF with 8.5 μl carbityl **chloroformate**. The **mixed anhydrides** were then conjugated with **glucose** 6-phosphate dehydrogenase [9001-40-5]. Hapten acid carbityloxycarbonyl esters and their **glucose** 6-phosphate dehydrogenase complexes, and hapten complexes with 2,2,5,5-tetramethyl-3-amino-1-oxylpyrrolidine were also prepared

L9 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1991:470186 CAPLUS
DOCUMENT NUMBER: 115:70186
TITLE: Polyol polyester preparation in aqueous media using mixed anhydrides and use of the polyesters as fat substitutes in food and in cosmetics
INVENTOR(S): Lalezari, Iraj
PATENT ASSIGNEE(S): USA
SOURCE: PCT Int. Appl., 19 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9101322	A1	19910207	WO 1990-US4029	19900718
W: AU, CA, JP				
RW: AT, BE, CH, DE, DK, ES, FR, GB, IT, LU, NL, SE				
US 5498708	A	19960312	US 1989-381208	19890718
AU 9061881	A1	19910222	AU 1990-61881	19900718
PRIORITY APPLN. INFO.:			US 1989-381208 A	19890718
			WO 1990-US4029 A	19900718

AB Partially or completely esterified polyols, e.g. sugars, for use as dietary fat substitutes or in cosmetics are prepared in an aqueous medium by the reaction of the polyol with a mixed anhydride of a carboxylic acid and an **alkyl chloroformate**. A mixed anhydride was prepared from myristic acid (0.02 mol) and Et chloroformate in the presence of triethylamine (over an ice bath). To this was added sucrose (0.005 mol) and an oil formed. After extraction with petroleum ether and drying, a sucrose myristate was recovered (yield 97.4% based on sucrose tetramyristate).

L9 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1991:470186 CAPLUS
DOCUMENT NUMBER: 115:70186
TITLE: Polyol polyester preparation in aqueous media using mixed anhydrides and use of the polyesters as fat substitutes in food and in cosmetics
INVENTOR(S): Lalezari, Iraj
PATENT ASSIGNEE(S): USA
SOURCE: PCT Int. Appl., 19 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9101322	A1	19910207	WO 1990-US4029	19900718
W: AU, CA, JP				
RW: AT, BE, CH, DE, DK, ES, FR, GB, IT, LU, NL, SE				
US 5498708	A	19960312	US 1989-381208	19890718
AU 9061881	A1	19910222	AU 1990-61881	19900718
PRIORITY APPLN. INFO.:			US 1989-381208 A	19890718
			WO 1990-US4029 A	19900718

AB Partially or completely esterified polyols, e.g. sugars, for use as dietary fat substitutes or in cosmetics are prepared in an aqueous medium by the reaction of the polyol with a mixed anhydride of a carboxylic acid and an **alkyl chloroformate**. A mixed anhydride was prepared from myristic acid (0.02 mol) and Et chloroformate in the presence of triethylamine (over an ice bath). To this was added sucrose (0.005 mol) and an oil formed. After extraction with petroleum ether and drying, a sucrose myristate was recovered (yield 97.4% based on sucrose tetramyristate).

ANSWER 1 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1993:255308 CAPLUS
DOCUMENT NUMBER: 118:255308
TITLE: Synthesis and biological activity of [L-hydroxyproline]3-tuftsin analog and its α- or β-O-D-glucosylated derivatives
AUTHOR(S): Biondi, L.; Filira, F.; Rocchi, R.; Tzehoval, E.; Fridkin, M.
CORPORATE SOURCE: Biopolym. Res. Cent., CNR, Padua, Italy
SOURCE: International Journal of Peptide & Protein Research (1993), 41(1), 43-51
CODEN: IJPPC3; ISSN: 0367-8377
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Syntheses are described of the Hyp3-tuftsin analog and of its derivs. α- or β-O-glycosylated at the side chain function of the hydroxyproline residue. The carbohydrate-free tetrapeptide was prepared by reacting Z-Thr-Lys(Z)-OH (Z = PhCH2O2C) with H-Hyp-Arg(NO2)-OBzl (Bzl = benzyl) by the **mixed anhydride** procedure. In the synthesis of the α-glycosylated analog, the O-glycosyl amino acid was incorporated by reacting Boc-(Glcα + β)Hyp-OH (Glc = D-**glucopyranosyl**) with H-Arg(NO2)-OBzl through the same procedure. The α- **glucosylated** dipeptide was isolated from the diastereomeric mixture, selectively deblocked, and **acylated** with Z-Thr-Lys(Z)-OH by the **mixed anhydride** procedure. In the preparation of the β- **glucosylated** analog, the BOP procedure was used for reacting Boc-[Glc(Ac)4β]Hyp-OH with H-Arg(NO)2-OBzl was well as for the final coupling to tetrapeptide. Removal of protecting groups from crude tetrapeptides was achieved by catalytic hydrogenation. Deacetylation of the sugar moiety of the β- **glucosylated** tetrapeptide was achieved by treatment with sodium methoxide in methanol. The synthetic compds. were isolated by ion exchange chromatog., and characterized by elemental anal., amino acid anal., optical rotation and proton NMR. Their capacity to evoke the release of interleukin 1 from mouse peritoneal macrophages and to modulate immunogenic activity of antigen-fed cells was evaluated, in comparison with tuftsin and rigin. All of the analogs were found to possess tuftsin-like activity.

L10 ANSWER 2 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1992:592210 CAPLUS
DOCUMENT NUMBER: 117:192210
TITLE: Syntheses of N-**acyl** and N-alkoxycarbonyl derivatives of 2-[(alkoxycarbonyl)amino]-2-deoxy-D-glucose.
AUTHOR(S): Lafont, Dominique; Boullanger, Paul
CORPORATE SOURCE: ESCIL, Univ. Lyon I, Villeurbanne, 69622, Fr.
SOURCE: Journal of Carbohydrate Chemistry (1992), 11(5), 567-86
CODEN: JCACDM; ISSN: 0732-8303
DOCUMENT TYPE: Journal
LANGUAGE: English
AB N-**Acyl** and N-alkoxycarbonyl derivs. of 1,3,4,6-tetra-O-acetyl-2-alkoxycarbonylamino-2-deoxy-β-D- **glucopyranose** were synthesized using **mixed anhydrides** and sym. or unsym. pyrocarbonates. These derivs. were used as donors in 1,2-trans-glycosylation reactions promoted by Lewis acids. Besides the expected β-D-glycosides, cyclization and rearrangement side-products were often encountered in such glycosylations.

L10 ANSWER 3 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1992:106696 CAPLUS
DOCUMENT NUMBER: 116:106696
TITLE: Preparation of N-**acylglycosylamines** as surfactants

INVENTOR(S): Plusquellec, Daniel; Pascale, Leon
PATENT ASSIGNEE(S): Ecole Nationale Superieure de Chimie de Rennes, Fr.
SOURCE: Fr. Demande, 10 pp.
CODEN: FRXXBL
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 2657611	A1	19910802	FR 1990-1317	19900201

PRIORITY APPLN. INFO.: FR 1990-1317 19900201
OTHER SOURCE(S): MARPAT 116:106696
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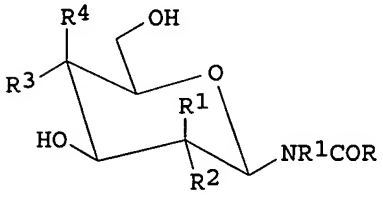

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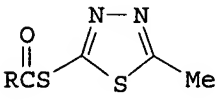

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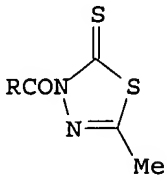

III

AB The title compds. [I; R = alkyl, aryl; R1 = H, alkyl, aryl; R2, R4 = H, OH; R3 = H, OH, **glucosyloxy**, galactosyloxy; with provisos], useful as surfactants (no data), are prepared by, e.g., reacting a **mixed anhydride** of the appropriate acid with glycosylamine, reacting a glycosylamine with (**acylthio**)methylthiadiazole II or **acylmethylthiadiazolinethione** III. ClCO2CH2CHMe2 and Et3N were added to a solution of decanoic acid in DMF, the solution was cooled at -5°. Sep., a solution of **glucosylamine** in DMF, obtained by heating the mixture to 60°, was cooled to ambient temperature and then added to the above anhydride suspension, and the resulting mixture was stirred for 16 h to give N-decanoylglucosylamine.

L10 ANSWER 4 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1989:173645 CAPLUS
DOCUMENT NUMBER: 110:173645
TITLE: New synthetic methods and reagents for complex carbohydrates. II. Synthesis of 2-**acylamino**-2-deoxy-D-**glucopyranose** derivatives by dimethylphosphinothioic **mixed anhydride** method
AUTHOR(S): Inazu, Toshiyuki; Hosokawa, Hideaki; Amemiya, Masahide
CORPORATE SOURCE: Noguchi Inst., Tokyo, 173, Japan
SOURCE: Bulletin of the Chemical Society of Japan (1988), 61(12), 4467-9
CODEN: BCSJA8; ISSN: 0009-2673
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 110:173645
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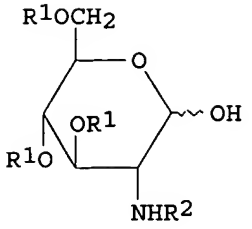

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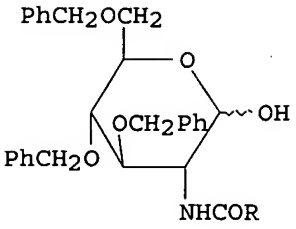

II

AB RCO2P(S)Me2 [R = Me, Ph, HOCH2, HO(CH2)2, HO(CH2)15], formed by treating RCO2H with Me2P(S)Cl in CH2Cl2 or THF in the presence of Et3N or (Me2CH)2NEt, reacted with aminoglucose I (R1 = PhCH2, R2 = H) to give 68-100% N-**acylated** derivs. II (R = same as above). Thus, treatment of I (R1 = R2 = H) with PhCO2P(S)Me2 and (Me2CH)2NEt in MeOH gave 76% I (R1 = H, R2 = Bz).

L10 ANSWER 5 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1988:510849 CAPLUS
DOCUMENT NUMBER: 109:110849
TITLE: Preparation and testing of glycosylamides as immunostimulants
INVENTOR(S): Lockhoff, Oswald; Hayauchi, Yutaka; Stadler, Peter; Brunner, Helmut
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.
SOURCE: Ger. Offen., 17 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.		DATE
DE 3604681	A1	19870820	DE 1986-3604681		19860214
US 4891425	A	19900102	US 1987-7703		19870128
EP 234348	A2	19870902	EP 1987-101462		19870203
EP 234348	A3	19890426			
EP 234348	B1	19910529			
R: AT, BE, CH, DE, ES, FR, GB, IT, LI, NL, SE					
AT 63920	E	19910615	AT 1987-101462		19870203
ES 2039211	T3	19930916	ES 1987-101462		19870203
CA 1271190	A1	19900703	CA 1987-529610		19870212
JP 62192397	A2	19870822	JP 1987-29921		19870213
PRIORITY APPLN. INFO.:			DE 1986-3604681	A	19860214
			EP 1987-101462	A	19870203

OTHER SOURCE(S): MARPAT 109:110849
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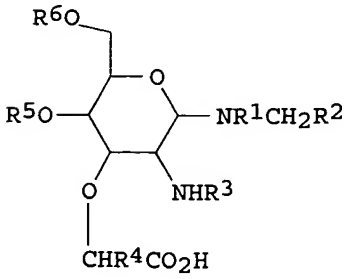

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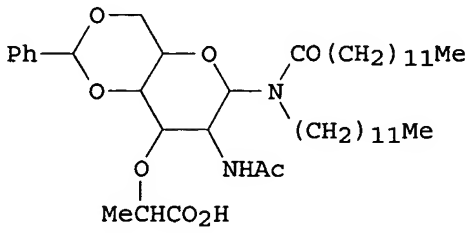

II

AB The title compds. [I; R1, R2 = H, (un)saturated C1-50 alkyl; R3 = H, **acyl**; R4 = H, alkyl; R5, R6 = H, **acyl**, alkyl, aralkyl; R5R6 = R7R8C; R7,R8 = H, alkyl, (substituted) aryl] were prepared as immunostimulants. 2-Acetylamino-2-desoxy-D-**glucose** was aminated with Me(CH2)11NH2, **acylated** with the **mixed anhydride** from Me(CH2)11CO2H and EtO2CCl, and ketalized with PhCH(OMe)2/tosic acid. The product was deprotonated with NaH in dioxane at 95°, and L-2-chloropropanoic acid was added at 60° to give amino sugar II. II at 10 mg/kg orally in rats reduced the number of Salmonella typhimurium in the blood to 4% of controls.

L10 ANSWER 6 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1988:6382 CAPLUS
DOCUMENT NUMBER: 108:6382
TITLE: Synthesis of modified tuftsins containing monosaccharides or monosaccharide derivatives
AUTHOR(S): Rocchi, R.; Biondi, L.; Filira, F.; Gobbo, M.; Dagan, S.; Fridkin, M.
CORPORATE SOURCE: Biopolym. Res. Cent., Univ. Padova, Padua, 35131, Italy
SOURCE: International Journal of Peptide & Protein Research (1987), 29(2), 250-61
CODEN: IJPPC3; ISSN: 0367-8377
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 108:6382
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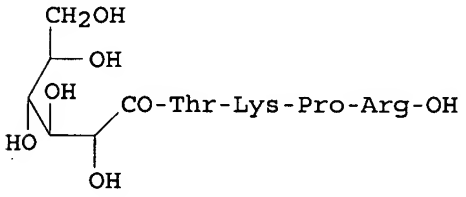

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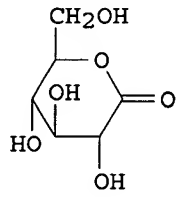


III

AB Nα- **Gluconyltuftsin** I was prepared by **acylating** H-Thr-Lys(Z)-Pro-Arg(NO2)-OBzl (II; Z = CO2CH2Ph, Bzl = CH2Ph) with D-**gluco**-1,5-lactone (III) and deblocking the resulting protected glycopeptide by hydrogenolysis. Boc-Arg(NO2)-OH (Boc = Me3CO2C) was condensed with 2-amino-2-deoxy-D-**glucopyranose** (H2NGlc) by the **mixed anhydride** method to give Boc-Arg(NO2)-NHGlc, which was used in the synthesis of H-Thr-Lys-Pro-Arg-NHGlc. Z-Thr[Glc(Bzl)4]-OC6H4NO2-o [Glc(Bzl)4 = 2, 3, 4, 6-tetra-O-benzyl-D-**glucopyranosyl**] (a mixture of α and β anomers) was coupled with II to give Z-Thr[Glc(Bzl)4-Lys(Z)-Pro-Arg(NO2)-OBzl (IV) as a mixture of α and β anomers, which were separated partially by flash chromatog. IV was deblocked by hydrogenolysis to give H-Thr(Glc)-Lys-Pro-Arg-OH.

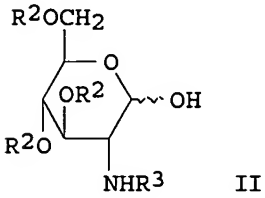
L10 ANSWER 7 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1987:617974 CAPLUS
DOCUMENT NUMBER: 107:217974
TITLE: Selective N-**acylation** of 2-amino-2-deoxy-D-glucopyranose derivatives without protecting the hydroxy functions
AUTHOR(S): Inazu, Toshiyuki
CORPORATE SOURCE: Noguchi Res. Inst., Tokyo, 173, Japan
SOURCE: Noguchi Kenkyusho Jiho (1986), (29), 17-21
CODEN: NOGUAR; ISSN: 0369-5131

DOCUMENT TYPE: Journal
LANGUAGE: Japanese
AB Dimethylphosphinothioyl **mixed anhydrides** of carboxylic acids [e.g., AcOP(S)Me2] are useful for N-**acylation** of 2-amino-2-deoxy-D-**glucopyranose** derivs. without protecting the hydroxy functions. This method also enables N-benzoylation of 2-amino-2-deoxy-D-**glucopyranose**.HCl in MeOH.

L10 ANSWER 8 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1987:407520 CAPLUS
DOCUMENT NUMBER: 107:7520
TITLE: Method for selective N-**acylation** of amino sugars
INVENTOR(S): Inazu, Toshiyuki
PATENT ASSIGNEE(S): Noguchi Research Institute, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 61197589	A2	19860901	JP 1985-38089	19850226
JP 05057993	B4	19930825		

PRIORITY APPLN. INFO.: JP 1985-38089 19850226
GI



II

AB Amino sugars were selectively **acylated** at NH2 groups by **mixed anhydrides** RCO2P(S)Me2 [I; R = (hydroxy)alkyl, aryl] in the presence of organic bases, e.g., Et3N, (Me2CH)2NEt, in common solvents, e.g., MeOH, THF, DMF, CH2Cl2. I were prepared in situ from (hydroxy) aliphatic acids or their salts and Me2P(S)Cl in the presence or absence (for acid salts) of organic bases. Thus, Me2P(S)Cl in THF was added dropwise to an ice-cooled solution of HOCH2CO2H in THF containing (Me2CH)2NEt and after 1/2 h, D-**glucosamine** derivative II.HCl (R2 = CH2Ph, R3 = H) and (Me2CH)2 were added. The resulting mixture was allowed to react at room temperature overnight to give 83% II (R2 = CH2Ph, R3 = HOCH2CO).

L10 ANSWER 9 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1984:34817 CAPLUS
DOCUMENT NUMBER: 100:34817
TITLE: Hydrophilic analogs of substance P: introduction of sugar acids and sulfonium groups
AUTHOR(S): Bienert, Michael; Forner, Klaus; Mehlis, Burkhard; Niedrich, Hartmut; Bergmann, Jutta; Kraft, Regine
CORPORATE SOURCE: Inst. Wirkstofforsch., Akad. Wiss. DDR, Berlin, DDR-1136, Ger. Dem. Rep.
SOURCE: Pept., Proc. Eur. Pept. Symp., 17th (1983), Meeting Date 1982, 517-20. Editor(s): Blaha, Karel; Malon, Petr. de Gruyter: Berlin, Fed. Rep. Ger.

CODEN: 50GFAA
DOCUMENT TYPE: Conference
LANGUAGE: English
AB Title analogs were prepared by coupling **gluconic** or galactonic acids with substance P (SP) or SP(7-11) [R-Phe-Phe-Gly-Leu-Met-NH2 (I; R = H)] via the active ester or **mixed anhydride** methods. The relative smooth muscle contracting potencies of **gluconyl** -SP(7-11) (I; R = HOCH2CH(OH)4CO), SP(7-11), and I [R = p-HOC6H4CH2CO (a hydrophobic residue)] are 0.1, 0.01, and 4.0, resp. Analogs containing hydrophilic sulfonium- and S-oxide structures at position 11 of C-terminal SP hexa- and pentapeptides exhibited only weak activity on smooth muscle.

L10 ANSWER 10 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1980:447209 CAPLUS
DOCUMENT NUMBER: 93:47209
TITLE: Polyhydroxycarboxylic acid peptides
INVENTOR(S): Forner, Klaus; Bienert, Michael; Niedrich, Hartmut
PATENT ASSIGNEE(S): Akademie der Wissenschaften der DDR, Institut fuer Werkstofforschung, Ger. Dem. Rep.
SOURCE: Ger. (East), 14 pp.
CODEN: GEXXA8
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DD 137836	Z	19790926	DD 1978-206891	19780724
PRIORITY APPLN. INFO.:			DD 1978-206891	A1 19780724

AB Title compds. HOCH2[CH(OH)]nCOR (n = 3,4; R = peptide moiety with 3 to 15 amino acid residues) were prepared Thus, substance P pentapeptide H-Phe-Phe-Gly-Leu-Met-NH2 was **acylated** with pentaacetylgalactonic acid and pentaacetylgluconic acid by active ester or **mixed anhydride** methods to give galacto- and **gluco**-AcOCH2[CH(OAc)]4CO-Phe-Phe-Gly-Leu-Met-NH2, which were deacetylated to give galacto- and **gluco**-HO[CH(OH)]4CO-Phe-Phe-Gly-Leu-Met-NH2.

L10 ANSWER 11 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1968:497042 CAPLUS
DOCUMENT NUMBER: 69:97042
TITLE: Selective **acylation** of D-glucose. Preparation of surface-active D-glucose 6-fatty acid esters
AUTHOR(S): Reinefeld, E.; Korn, H. F.
CORPORATE SOURCE: Tech. Univ. Braunschweig, Brunswick, Fed. Rep. Ger.
SOURCE: Staerke (1968), 20(6), 181-9
CODEN: STRKA6; ISSN: 0038-9056
DOCUMENT TYPE: Journal
LANGUAGE: German
AB **D-Glucose** was partially esterified with different **acylating** reagents (acid chloride, acid imidazolide, **mixed anhydride** of fatty acid, and Et ester of carbonic acid) and in different ratios with lauric acid. In any case D-**glucopyranose** 6-ester, D-**glucopyranose** 1,6-diester and D-**glucopyranose** 2,6-diester were found as coexisting reaction products. As in the case of p-tolysulfonylation, the primary alc. group is esterified most readily, and therefore the formation of 6-ester is favored. The ratios of diester isomers, which were isolated by preparative thin-layer chromatog., indicate that after the 6-position, the anomeric hydroxyl and the 2-hydroxyl are **acylated** most readily. Investigations on D-**glucose** derivs. showed that the course of the partial **acylation** is not substantially affected when one of these two

hydroxyl groups is blocked. In the case of the **mixed anhydride**, the smallest yield of diesters results, besides the monoester. From D-**glucose** and the fatty acid chlorides, a homologous series of surface active D-**glucopyranose** 6-esters of the natural fatty acids from caproic acid to steric acid were prepared D-**Glucopyranose** 6-laurate has the greatest surface activity (σ = 29.4 dyne/cm., 0.001M solution).

L10 ANSWER 12 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1959:94834 CAPLUS
DOCUMENT NUMBER: 53:94834
ORIGINAL REFERENCE NO.: 53:17142h-i,17143a-d
TITLE: Synthesis of N-aminoacyl derivatives of aminopyrimidines and 3-β-D-**glucopyranosylcytosine** by the action of **mixed anhydrides** of phosphoric acids and amino acids
AUTHOR(S): Prokof'ev, M. A.; Bogdanov, A. A.
CORPORATE SOURCE: M. V. Lomonosov State Univ., Moscow
SOURCE: Nauchnye Doklady Vysshei Shkoly, Khimiya i Khimicheskaya Tekhnologiya (1959), (No. 1), 134-7
CODEN: NDVSAJ; ISSN: 0470-469X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB To a solution of 1.23 g. 6-amino-2,4-dimethylpyrimidine (I) in 40 ml. dry dioxane was added 4.7 g. of the **mixed anhydride** of dibenzylphosphoric acid and carbobenzoxyglycine (II). The mixture stirred 2 hrs. at room temperature, kept overnight at 37°, the mixture evaporated in vacuo, the residue treated with CHCl3, the CHCl3 solution washed with H2O, saturated NaOH solution twice, washed again with H2O, dried (Na2SO4), evaporated in vacuo, 30 ml. anhydrous ligroine added to the residual oil, and the precipitate filtered off gave 28.6% 6-carbobenzoxyglycylamino-2,4-dimethylpyrimidine (III), m. 114-17°, λ 263 mμ (ε 7100). To a solution of 2.09 g. II in 40 ml. dried dioxane was added 1.01 g. Et3N and 1.47 g. dichloroethyl phosphite. The Et3N.HCl filtered off, a solution of I in 40 ml. dry dioxane added to the filtrate, the mixture heated 15 min. at 105-10°, kept overnight at room temperature, the solution evaporated in vacuo, the residual oil dissolved in 30 ml. CHCl3, the CHCl3 solution washed with H2O, twice with 2N NaOH, and then with H2O, dried (Na2SO4), evaporated in vacuo, 20 ml. ligroine added to the residue, and the precipitate filtered off yielded 46% III, m. 115-17°. 2-Carbobenzoxyalanylamino-6-oxo-1,4-dimethylpyrimidine, m. 127-8°, λ 270 mμ (ε 10,100) was obtained in 65% yield as for III (2nd procedure) from 6-oxo-2-amino-1,4-dimethylpyrimidine and carbobenzoxyalanine. 6-Carbobenzoxyleucylamino-2,4-dimethylpyrimidine, m. 67-70°, λ 263 mμ (ε 5500) was similarly obtained in 40% yield from I (2nd procedure) and carbobenzoxyleucine (IV). N-Carbobenzoxyglycyl-3-β-D-tetraacetylglucopyranosylcytosine (V), m. 117-20°, λ 250, and 300 mμ (ε 8200, 3300) was similarly obtained in 5% yield (1st procedure) from 3-β-D- **glucopyranosylcytosine** (VI) and II. N-carbobenzoxyleucyl-3-β-D-tetraacetylglucopyranosylcytosine, m. 129-32° (decomposition), λ 250 and 302 mμ (ε 8100, 3000) was similarly obtained (1st procedure) from IV and VI. V (3 mg.) was boiled with 1 ml. 0.1N NaOH 1 hr., the solution neutralized by N HCl and the hydrolysis products chromatographed in 5% aqueous Na2HPO4 saturated with iso-AmOH. A stain of VI (Rf 0.88) was observed under ultraviolet light and a stain of II (Rf 0.86) by wetting the paper with a mixture of aqueous solns. of starch, 10% KI and 0.5% KIO3 (1:1:1).

L10 ANSWER 13 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1959:11839 CAPLUS

DOCUMENT NUMBER: 53:11839
ORIGINAL REFERENCE NO.: 53:2239a-i,2240a-b
TITLE: Aminoacyl derivatives of a few nucleosides and nucleotides
AUTHOR(S): Prokof'ev, M. A.; Shabarova, Z. A.; Sokolova, N. I.
SOURCE: Vestnik Moskovskogo Universiteta, Seriya Matematiki, Mekhaniki, Astronomii, Fiziki, Khimii (1957), 12(No. 6), 215-24
CODEN: VMUMAB; ISSN: 0579-9376
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
GI For diagram(s), see printed CA Issue.
AB Aminoacyl and peptide derivs. of 3-(β-D- **glucopyranosyl**)cytosine were synthesized by 3 different ways: (1) the aminoacyl derivs. of the nucleoside were converted in good yield on heating into the 3-(β-D-tetraacetylglucopyranosyl)cytosine-HCl (I) with the HCl salts of phthalylamino acids in dioxane; (2) carbobenzoxy-substituted amino acids or peptides and I in the presence of N,N'-dicyclohexylcarbodiimide in the cold in dioxane yielded N6-aminoacyl and N6-peptide derivs. of cytosine nucleoside; this method is recommended for preparation purposes; (3) by **acylation** of **glucopyranosylcytosine** (II) with **mixed anhydrides** of carbobenzoxyamino acids and peptides. The synthesized N6-carbobenzoxyaminoacyl and -peptide derivs. of 3-β-D-II, OC.N:C(NHR).CH: CH.NZ (Z = tetraacylglucopyranosyl), and their properties were [carbobenzoxy-substituted R, **acyl**, method, % yield, m.p. (decomposition), and λ (mμ) given]: phthalylglycyl, Ac, 1 and 3, 60, 152-5°, 240 and 290; phthalylglycyl, phthalylglycyl, 1, 40, 239-40°, 297; glycyl, Ac, 1 and 3, 54, 119-21°, 250 and 300; valyl, Ac, 3, 40, 92°, 250 and 300; leucyl, Ac, 2, 18, 145-6°, 250 and 300; phenylalanyl, Ac, 2 and 3, 81,143-5°, 250 and 300; valylglycyl, Ac, 3, 40, 120-5°, 250 and 300; valylleucyl, Ac, 3, 15, 120° 250 and 300; valylphenylalanyl, Ac, 3, 40, 128-30°, 250 and 300; phenylalanylglycylglycyl, Ac, 2, 42, 139-40°, 250 and 300; valylglycylphenylalanyl, Ac, 2, 38, 106-8°, 250; valylleucylphenylalanyl, Ac, 2, 32, 110°, 250; valylphenylalanylphenylalanyl, Ac, 2, 46, 103°, 250. The hydrolysis of a few of the above substances was carried out (**acyl**, hydrolyzing agent (at 100°), min. time (in min.) to attain 100% hydrolysis of the amide bond given): glycyl, 0.1N NaOH, 60 (2N HCl, 20); phenylalanyl, 0.1N NaOH, 80 (N HCl, 15); valyl, 0.1N HCl, 50 (N HCl, 60; 2N HCl, 20); valylglycyl, 0.1N NaOH, 30 (2N HCl, 60); valylphenylalanyl, 0.1N NaOH, 50 (N HCl, 60); valylleucyl, 0.1N NaOH, 60 (2N HCl, 20). It is noted that N6-carbobenzoxyaminoacyl derivs. of cytosine nucleoside are stable toward the hydrolytic action of water (even after 50 hrs. boiling). There is a sharp difference in the hydrolysis of these compds. and the aminoacyl derivs. of nucleosides when the protecting carbobenzoxy group is absent; this group is removed by reduction with H on Pd dust in dioxane at 70-80°. These derivs. (**acyl** = Ac) are listed [R, % yield, m.p. (decomposition), λ (mμ)]: glycyl, 76, 86-8°, 250; phenylalanyl, 92, 92-4°, 250; valylglycylphenylalanyl, 72, 94-7°, 250; valylleucylphenylalanyl, 60, 98-100°, 250; valylphenylalanylphenylalanyl, 68, 106-8°, 250. The removal of the carbobenzoxy group is accompanied by an increase in the lability of the amide bonds and consequently an increase in the ease with which they are hydrolyzed. The amino groups in adenosine and 9-(β-D-**glucopyranosyl**)guanine (III) show a remarkable inertness toward the **acylation** agents and the synthesis of the aminoacyl derivs. by means of the usual methods, even with variations in temps., and solvents, was unsuccessful. The only way of obtaining these derivs. is to use the chloroanhydride of the phthalylated amino acids. In the case of triacetyladenosine, the reaction was carried out with chloroanhydrides of phthalylated amino acids in absolute C6H6 in the presence of Bu3N by boiling a few hrs. (pyridine can be employed as solvent). If free adenosine or III is brought into the reaction, the corresponding

tetrakis(phthalylaminoacyl) derivs. are formed. The following adenosine derivs. were prepared [N6-amino substituent, **acyl** group, % yield, m.p. (decomposition), λ (mμ), and Rf(BuOH saturated with water) given]: phthalylglycyl, Ac, 51, 118-20°, 266-8, 0.55; phthalylglycyl, phthalylglycyl, 61, 230°, 260-2, -; phthalylvalyl, Ac, 55, 95-100°, 266, 0.47; phthalylphenylalanyl, Ac, 50, 105-9°, 266, 0.62. The following N2-amino derivs. of 9-(β-D-**glucopyranosyl**)guanine were prepared [N2-amino substituent, **acyl** group, % yield, and m.p. (decomposition) given]: phthalylglycyl, phthalylglycyl, 78, 155°; phthalylvalyl, phthalylvalyl, 65, 125°; phthalylphenylalanyl, phthalylphenylalanyl, 40, 165°. The stability of the amide bond in these compds. toward hydrolysis was investigated. Lengthy boiling (up to 50 hrs.) with water did not promote the hydrolysis of the bond. Both the amide and the complex ester bonds hydrolyzed after 30 min. when boiled with 0.1N NaOH. Preliminary results on the synthesis of aminoacyl derivs. of cytidylic acid nucleotides have been obtained. By the interaction of chromatographically pure cytidylic acid with a mixture of the anhydride of carbobenzoxy phenylalanine and H2CO3, a new spot appeared on the chromatogram. The maximum absorption of this spot is characteristic of N6-aminoacylated derivs. of cytosine (250 mμ). It is supposed that the formation of N6-carbobenzoxyphenylalanyl derivative of cytidylic acid took place.

L10 ANSWER 14 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1958:83029 CAPLUS
DOCUMENT NUMBER: 52:83029
ORIGINAL REFERENCE NO.: 52:14726a-d
TITLE: Cycloborate esters of 16α,17α-dihydroxysteroids
INVENTOR(S): Thomas, Gordon H.
PATENT ASSIGNEE(S): Olin Mathieson Chemical Corp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2831003		19580415	US 1957-694527	19571105

AB Cycloborate esters of 16α,17α-dihydroxy-3,20-dioxo-4-pregnenes and 1,4-pregnadienes, which may be further substituted in positions 2 and 6 by Me, in 9 or 12 by a halogen, OH, or alkoxy, in 11 by oxo or OH, and in 21 by a halogen, OH, or **acyloxy** group, are prepared by treating the 16α,17α-diol with B2O3. The products can be converted into salts, esters, or into **mixed anhydrides**, e.g. with Ac2O. Thus, 200 mg. 9α-fluoro-4-pregnene-11β,16α,17α,21-tetrol-3,20-dione and 1 g. B2O3 in 5 mL. MeOH is refluxed 1 h., diluted with 30 mL. H2O, and the precipitate crystallized (Me2CO-hexane) giving 155 mg. 16α,17α-cycloborate ester (I), m. above 300°, λ 238 mμ (alc.) (ε 16,000), 2.9, 5.82, 6.10 μ (Nujol), neutralization equivalent 415. N NaOH (1.1 mL.) is added to 500 mg. I in 20 mL. cooled MeOH under N and the solvent evaporated in vacuo giving the Na salt of I, which (500 mg.) in 20 mL. MeOH is refluxed 4 h. with 3 mL. MeI, the solution concentrated, H2O added, and the Me ester of I extracted with CHCl3. Similarly are prepared the 16α,17α-cycloborate esters of 9α-fluoro-1-4-pregnadiene-11β,16α,17α,21-tetrol-3,20-dione (II), 16α-hydroxy-hydrocortisone, 16α-hydroxyprednisolone, 12α-fluoro-16α-hydroxyhydrocortisone, and 12α-fluoro-16α-hydroxyprednisolone, also the Na salt and Me ester of II. The compds. display **glucocorticoid** and antiinflammatory activity and can be used for treatment of rheumatoid arthritis.

L10 ANSWER 15 OF 18 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1956:77523 CAPLUS
DOCUMENT NUMBER: 50:77523
ORIGINAL REFERENCE NO.: 50:14541a-i,14542a-i,14543a-i,14544a-e
TITLE: Synthetic emulsifying agents
AUTHOR(S): Fieser, Mary; Fieser, Louis F.; Toromanoff, Edmond; Hirata, Yoshimasa; Heymann, Hans; Tefft, Melvin; Bhattacharya, Sivaprasad
CORPORATE SOURCE: Harvard Univ.
SOURCE: Journal of the American Chemical Society (1956), 78, 2825-32
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB C18H37OH (10.8 g.) in 180 cc. CHCl3 added slowly with vigorous stirring and cooling to 6 cc. PhP(O)Cl2 in 16 cc. CHCl3 and 3.4 cc. pyridine, the mixture warmed 10 min. at 35°, treated with 5.6 g. dry powdered HO(CH2)2NMe3Cl, stirred 48 hrs. at room temperature, and evaporated, the residue extracted with three 50-cc. portions Et2O, the insol. residue dissolved in 50 cc. H2O, the solution saturated with NaCl and extracted with CHCl3, and the extract evaporated yielded 7 g. C18H37OP(O)(OPh)OCH2CH2NMe3Cl (I), m. 82-6° (from Me2CO). I (3.0 g.) hydrogenated in EtOH over PtO2 yielded 1.8 g. C18H37OP(O)(OH)OCH2CH2NMe3Cl (Ia) m. 71-2° (from Me2CO). Ia in EtOH treated with Amberlite IRA-400, the solvent partially removed, the residue diluted with Me2CO, and the crude precipitate chromatographed and eluted with 4:1 CHCl3-EtOH gave the corresponding hydroxide, m. 220-30°; it is sparingly soluble in H2O and Nujol at room temperature and shows no emulsifying properties. Dihydrophytyl and cholestanyl phosphorylcholine were prepared in essentially the same manner but could not be obtained pure; the crude dihydrophytyl derivative (semisolid) showed some emulsifying action. L-Arabinose (75 g.) in H2O treated at room temperature 12 hrs. with 120 g. Br, the excess Br removed in vacuo at 40-50°, the mixture treated with 120 g. PbO, the white precipitate filtered off after several hrs., the filtrate treated dropwise with H2SO4 and filtered, concentrated in vacuo at 50°, and the residue diluted with 75 cc. MeOH and allowed to stand a few hrs. at 5° deposited 77% arabonolactone (II), m. 148-50° (from MeOH), αD30 -6.5°. II (2 g.) in MeOH treated with 2.2 g. C12H25NH2 and kept at room temperature deposited 87.5% N-laurylarabonamide, m. 150-1° (from EtOH or dioxane). Similarly were prepared the following N-alkylarabonamides (alkyl group and m.p. given): C10H21, 150-1° (from EtOH); C14H29, 150-1° (from EtOH); C16H33, 150-1° (from dioxane); C18H37, 149-50° (from dioxane). **Gluconolactone** condensed with C18H37NH2 (IIa) at 140° or in refluxing EtOH during 1 hr. gave N-stearylgluconamide, m. 149.4-54.8° (from EtOH). Similarly were prepared the following N-alkylgluconamides (III) (alkyl group and m.p. given): C12H25, 153.2-5.6°; C16H32, 150.4-4.6°. **Glucoheptonolactone** (2.08 g.), m. 148-52°, and 2.69 g. IIa gave similarly 55% N-stearylglucoheptonamide, m. 149-52° (cloudy)(from EtOH); it decomposed at about 180°. The C14-, C16-, and C18-III gave a solubility of about 6 g./l. boiling H2O; when used with cholesterol or the mono-stearyl ether of (CH2OH)2 emulsions with an average particle size of 5-10 μ can be obtained in a Waring Blendor; these emulsions are stable only for a few hrs. 1,2-Isopropylideneglucuronolactone (IV) was prepared in 81% yield by the method of Owen, et al. (C.A. 35, 6240.2), except that the volume of Me2CO was reduced to 500 cc. for 20 g. IV and Na2CO3 was used instead of BaCO3. IV (6.6 g.) in 50 cc. dioxane and 15 cc. cold concentrated NH4OH kept 4-5 hrs. in the cold room, and the solution evaporated in vacuo below 45° gave almost 100% 1,2-isopropylideneglucuronamide (V), needles,

m. 163-4° (from absolute EtOH), αD18 -13.5° (c 1, H2O). IV (5.8 g.) in 50 cc. dry tetrahydrofuran treated with 6.8 g. IIa in small portions with stirring, kept overnight in the cold room, and then a few hrs. at room temperature, the solvent removed in vacuo below 40° to incipient crystallization, and the residue diluted with petr. ether gave 8.0 g. 1,2-isopropylidene-N-stearylglucuronamide (VI), m. 92-3°; 2nd crop, 2.3 g., m. 86-90°. Similarly were prepared the following 1,2-isopropylidene-N-alkylglucuronamides in 70-90% yield (alkyl group, m.p., and αD in MeOH given): C10H21, 70-5° (from petr. ether), -14° (c 1.162); C12H25, 87-8° (from MeOH), -13° (c 1.046); C14H29, 88-90° (from MeOH), -12.5° (c 1.09); C16H33, 90-2° (from EtOH), -13.5° (c 1.064). ω-Cyclohexyldecanoic acid (10 g.) refluxed 2 hrs. with 15 cc. SOCl2 and evaporated in vacuo, and the cooled residue poured slowly into 100 cc. ice-cold concentrated NH4OH yielded 9 g. ω-cyclohexyldecanamide (VII), m. 89-93° (from aqueous MeOH). VII (7.6 g.) reduced in the usual manner with LiAlH4 in refluxing Et2O and the Et2O solution treated with HCl gave 6.3 g. ω-cyclohexyldecylamine HCl salt, m. 151-3° (from MeOH); free base, m. above 50°. The free amine in Et2O (liberated with aqueous NaHCO3 from the HCl salt) treated with IV gave 1,2-isopropylidene-N-(ω-cyclohexyldecyl)glucuronamide, m. 88-90°. V (2.3 g.) in 20 cc. H2O and 0.5 cc. concentrated HCl heated 1-3 min. at 80°, the H2O removed in vacuo, and the residue crystallized from absolute MeOH gave 1.8 g. glucuronamide (VIII).H2O, m. 168-9° (decomposition), αD22 70° → 31.9° (44 hrs., c 1.77, H2O); anhydrous VIII, m. 173-4°. γ-Lactone of β-methylglucuronoside (4.2 g.) in 20 cc. cold dioxane treated overnight with 10 cc. ice cold NH4OH (d. 0.9), the solvent removed in vacuo below 40°, and the residue hydrolyzed with HCl gave 2.1 g. VIII.H2O. VI (5 g.) in 100-350 cc. H2O and 7 cc. concentrated HCl tested with stirring 30-45 min. on the steam bath and the mixture cooled gave the corresponding N-alkylglucuronamides (alkyl group, m.p., and αD in MeOH given): C10H21, 145-8° (decomposition) (from aqueous MeOH), 24° (c 1.11); C12H25, 160-1° (from aqueous dioxane), -4° → 22° (24 hrs., c 1.18); C14H29, 156-7° (from aqueous dioxane), 11° → 24° (24 hrs., c 1.05); C16H33 (IX), 155-7° (from aqueous dioxane), 24.7° → 26° (24 hrs., c 1.03); C18H37 (X), 153-4° (from aqueous dioxane), 23° (10 min., c 1.046); ω-cyclohexyldecyl (XI), 128-30° (from MeOH), 21° → 25° (24 hrs., c 1.15). ω-Cyclohexylbutyramide, m. 103-6°, reduced to the amine (HCl salt, m. 165-7°), condensed with V, and the product hydrolyzed yielded 80% ω-cyclohexylbutylglucuronamide, m. 160-3° (from aqueous MeOH), αD 35.8° → 23.5° (24 hrs., c 1.54, MeOH). IX, X, and XI gave fairly stable oil-in-water emulsions when used with a co-emulsifier. IIa (2.5 g.) in 15 cc. cold tetrahydrofuran added to 2 g. β-methylglucuronoside-γ-lactone in cold tetrahydrofuran, the mixture kept overnight in the cold room and then 1-2 hrs. at room temperature, and the solvent removed in vacuo yielded 77% N-stearylamide (XII) of β-methylglucuronoside (XIII), m. 75-8° (from Et2O), αD21 -60.4° (c 1.03, MeOH). A similar run carried out at an initial temperature of 40-50° for 0.5 hr. and then at 25° for 2-3 hrs. yielded 87% higher melting form of XII, m. 93-5° (from MeOH-C6H6), αD25 -60.7° (c 1.0, MeOH). Similarly were prepared the following N-alkylamides of XIII (alkyl group, m.p., and αD in MeOH of form A and B given): C12H25, 68-70°, -58.4° (c 1.05), 88-90°, -58.7° (c 1.43); C14H29, 70-3°, -60.8° (c 1.11), 88-90°, -61° (c 1.04); C16H33, 75-8°, -60.6° (c 1.3), 92-3°, -60.5° (c 1.3). The glucuronosides were hydrolyzed with 1 cc. concentrated HCl in 100 cc. H2O to the corresponding glucuronamides in nearly 100% yield. The appropriate glucuronamide (5 g.) in 250-500 cc. hot H2O treated at 50-60° with 4 cc. Br at 40-50°, the solution kept in the cold room overnight, the excess Br removed with saturated aqueous Na2S2O3, and the

product air-dried and recrystd. from tetrahydrofuran gave about 80% of the corresponding N-alkylglucosaccharonamide (XIV) (alkyl group, m.p., and αD in tetrahydrofuran given): C12H25, 134-7°, -21.5° (c 1.13); C14H29, 125-7°, -22° (c 1.06); C16H33, 135-8° with previous sintering, -21° (c 1.14); C18H37, 137-9°, -22° (c 1.12). The XIV gave less stable emulsions than the corresponding glucuronamides; they are slightly more H2O-soluble C11H23COCl (2.2 g.) in 20 cc. tetrahydrofuran added dropwise with stirring to 2.15 g. **glucosamine**.HCl salt and 2 g. NaHCO3 in 20 cc. H2O with agitation, the mixture agitated 0.5 hr. and diluted with 100 cc. H2O, and the precipitate washed with H2O and recrystd. from dioxane-EtOH gave 3.2 g. N-lauroylglucosamine, m. 190-3°. Similarly were prepared the following N-**acylglucosamines** (XV) (**acyl** group and m.p. with decomposition given): C13H27CO, 193-5° (from dioxane-EtOH); C15H31CO, 190-3° (from dioxane-EtOH); C17H35CO, 190-1° (from dioxane-EtOH). C17H35CO2H (XVI) (11.4 g.) and 6 cc. Et3N in dry tetrahydrofuran treated with stirring and cooling at -5° with 4 cc. ClCO2Et and then after 5 min. without further cooling with the Na salt of 3.6 g. β-alanine in 30 cc. cold H2O, the mixture stirred 0.5 hr., acidified to pH 3-4, and filtered, and the residue washed with warm H2O, dried, extracted with petr. ether, and recrystd. from 4:1 dioxane-H2O or tetrahydrofuran yielded 11.2 g. stearoyl-β-alanine (XVII), m. 122-4°, insol. in H2O at 25°, somewhat soluble at 100°. In the same manner was prepared oleoyl-β-alanine (XVIII), m. 75-6° (from aqueous dioxane). XVIII (1 g.), 1.2 g. AgOAc, and 13 cc. glacial AcOH containing 0.1 cc. H2O treated during 40 min. with 0.72 g. iodine, the mixture heated 3 hrs. on the steam bath, cooled, filtered, and evaporated, the residue in MeOH refluxed 25 min. with aqueous KOH and filtered, and the filtrate acidified gave 0.6 g. 9,10-dihydroxystearoyl-β-alanine, m. 148-50° (from EtOH). XVII was converted in the usual manner in 71% yield to stearoyl-β-alanyl-β-alanine, m. 153-6° (from aqueous dioxane). Similarly were prepared: stearoyl-β-alanylglycine, 75%, m. 172-4° (from dioxane-H2O); stearoyl-β-alanyltaurine, 78%, m. about 200° (decomposition) (it contains solvent of crystallization which is not removed by drying at 150°). XVI (3 g.), 1.07 g. Et3N, and 1.44 g. ClCO2CH2CHMe2 in CHCl3-EtOAc treated with 1.62 g. α-alanine Et ester (XVIIIa) HCl salt and 1.07 g. Et3N gave 2.87 g. stearoyl-α-alanine Et ester (XIX), m. 62-5° (from ligroine). XIX (1 g.) in 10 cc. dioxane hydrolyzed with 3 cc. concentrated HCl in 1.5 cc. H2O on the steam bath during 1 hr. yielded 0.63 g. DL-stearoyl-α-alanine (XX), m. 115-17° (from ligroine-dioxane). XX and XVIIIa were converted by the **mixed anhydride** method to stearoyl-α-alanyl-α-alanine Et ester, m. 82-3°, which was hydrolyzed to the free acid, m. 132-3° (from petr. ether-dioxane). Similarly were prepared the following compds. (% yield and m.p. given): stearoylglycine (XXI), 75-80, 125-7° (from EtOAc-tetrahydrofuran); stearoylglycyl-β-alanine, 70-5, 169-70° (from dioxane); stearoylglycylglycine, 75-80, 170-2° (from dioxane); stearoylglycyltaurine, 80-90, -(practically insol. in various organic solvents; it crystallized from H2O with H2O of crystallization which is not lost by drying at 150°); stearoyltaurine, 73, m. about 240° (decomposition); stearoyl-DL-asparagine (XXII), 70, 145-8° (from dioxane); stearoylglycylasparagine (XXIII).H2O, 70-5°, 180-5° (from aqueous dioxane). XXII (0.4 g.) in 10 cc. dioxane treated with 0.08 g. NaNO2 in 30 cc. H2O, warmed 4-6 hrs. on the steam bath with 0.4 cc. concentrated HCl, and cooled to room temperature deposited 0.37 g. stearoyl-DL-aspartic acid (XXIV), m. 111-13° (from aqueous dioxane or EtOAc). XXIV heated 15 min. at 70-80° in Ac2O and cooled gave 100% stearoyl-DL-aspartic anhydride, m. 124-5° (from ligroine containing some tetrahydrofuran). Stearoyl-L-glutamic acid, m. 127-8° (from tetrahydrofuran), αD22 8.5° (c 1.62, dioxane), was prepared in

55% yield by the **mixed anhydride** method from L-glutamic acid and then converted in the usual manner to the anhydride, m. 107-9° (from ligroine-tetrahydrofuran). XXIII hydrolyzed with acid in the presence of NaNO2 yielded 80-90% stearoyl-DL-aspartic acid, m. 165-70°; also prepared in 40-60% yield directly from XXI; the acid was converted in the usual manner to the anhydride, m. 175-80°. C18H32CHBrCO2H (10 g.) heated 24 hrs. with excess 27% NH4OH in a pressure bottle and the product washed with H2O and boiling MeOH and ligroine gave 8.5 g. C16H33CH(NH2)CO2H (XXV), m. 223-4° (decomposition). XXV heated with phthalic anhydride 0.5 hr. at 145-60° gave the phthalimido derivative (XXVI) of XXV, m. 81° (from ligroine). XXVI (2 g.) refluxed 3 hrs. with 10 cc. SOCl2, the excess SOCl2 removed with suction, the residual oil washed with dry PhMe, dried at 1 mm., dissolved in 20 cc. dry CHCl3, and treated with 0.71 g. Et ester of α-alanine HCl salt in 10 cc. dry CHCl3, the mixture cooled to -20°, treated with stirring during 40 min. with 1.1 g. Et3N in dry CHCl3, warmed to room temperature, and evaporated in vacuo, and the residue dissolved in ligroine, washed with H2O, evaporated, and diluted with petr. ether yielded 0.9 g. Et ester (XXVII) of α-phthalimidostearoyl-α-alanine (XXVIII), crystals, m. 63-4°; XXVIII, m. 116° (from ligroine). XXVIII (0.45 g.) in 7 cc. 95% EtOH refluxed 45 min. with 1.5 cc. N2H4 and a few drops H2O, cooled, and diluted with H2O gave 0.28 g. α-aminostearoyl-α-alanine, m. 218-20°. N-Carbobenzyloxy-DL-alanine (4.46 g.), m. 120-2° in 50 cc. tetrahydrofuran containing 3 cc. Et3N treated with stirring at -5° with 5.4 g. IIa in 50 cc. tetrahydrofuran, the mixture stirred 0.5 hr. without cooling and acidified, the solvent partially removed in vacuo, the residue diluted with cold H2O, and the precipitate washed with cold dilute NH4OH and recrystd. from MeOH yielded 8 g. N-carbobenzyloxy-DL-alanylstearylamine (XXIX), m. 106-9°. XXIX (4.7 g.) in 100 cc. absolute MeOH hydrogenated overnight over 0.25 g. 10% Pd-C, filtered, and evaporated, and the residue heated a few hrs. at 80-90° gave DL-alanylstearylamine (XXX), m. 76-8° (from MeOH). Similarly were prepared the following dipeptides (m.p. and m.p. of the N-carbobenzyloxy derivative given): L-isomer of XXX, 70-3° (from Et2O), 103-4° (from MeOH); L-alanylcetylamine.0.5 H2O, 58-60° (from Et2O), m. 90-30 (from MeOH); L-alanyl-ω-cyclohexyldecylamine, 56-8° (from MeOH), 115-16° (from MeOH); L-leucylstearylamine, 66-8° (from MeOH), (hemihydrate) 96-8° (from MeOH); L-leucylcetylamine, 58-60° (from MeOH), 95-7° (from MeOH); L-prolylstearylamine, 70-2° (from MeOH), 88-90° (from MeOH); glycylstearylamine hemihydrate, 96-8° (from MeOH), 116-18° (from tetrahydrofuran); glycylcetylamine, 84-6° (from MeOH), 110-11° (from MeOH); β-alanylstearylamine hemihydrate, 85-7°, 124-6° (from tetrahydrofuran-MeOH) [carbamate, m. 126-7° (from MeOH)]; β-alanylcetylamine hemihydrate, 84-6° (from Et2O), 124-6° (from dioxane-MeOH) [carbamate, m. 112-14° (from MeOH)]. N-Carbobenzyloxy-L-cysteinylstearylamine, m. 156-61° (from tetrahydrofuran) reduced with Na in liquid NH3 yielded 40% N-cysteinylstearylamine, m. 74-6°. N-Carbobenzyloxyaspartic acid anhydride (7.56 g.) in 35 cc. PhCH2OH treated 1 hr. with cooling with 1 equivalent PhCH2ONa yielded 7 g. PhCH2OCONHCH(OCOCH2Ph)CH2CO2H which condensed with IIa via the **mixed anhydride** with ClCO2Et gave the dicarbobenzyloxy derivative of N-stearyl-L-asparagine (XXXI), m. 92-4° (from MeOH); this treated with MeOH with H over Pd-C gave 60% XXXI, m. 168-70° (from MeOH). N-Carbobenzoyloxy-L-alanine condensed with L-alanylstearylamine followed by hydrogenolysis gave 80% L-alanyl-L-alanylstearylamine, m. 115-17° (from MeOH); N-carbobenzyloxy derivative, m. 163-4° (from tetrahydrofuran and MeOH). Similarly was prepared β-alanyl-β-alanylstearylamine monohydrate, m. 160-3°; carbobenzyloxy derivative, m. 175-8°. (CH2OH)2 (84 cc.), 1.5 g. Na, 20 g. C18H37Br, and 10 cc. tetrahydrofuran heated 96 hrs. at 120°, cooled, diluted with H2O, and extracted with Et2O gave 4.3 g. distearyl ether of (CH2OH)2, m. 55-7°; concentration of the mother liquors

yielded 11.7 g. monostearyl ether (XXXII) of (CH2OH)2, white flaky solid, m. 51-2°. C18H37O(CH2)2CO2H (XXXIII) treated with LiAlH4 gave a product contaminated with C18H37OH (XXXIV). XXXIV (27 g.) added to 13 g. CH2:CHCO2Me in dry dioxane containing a trace of piperidine and PhCH2NMe3Br, the mixture refluxed overnight, concentrated, and diluted with H2O, the crude product washed with H2O and refluxed with 8 g. KOH in 500 cc. H2O, filtered, and acidified, the precipitate dissolved in Et2O, the solution treated with gaseous NH3, and the precipitate dissolved in H2O and acidified gave 4.5 g. XXXIII, m. 75-8° (from Et2O). XXXIII (1.7 g.) in dry tetrahydrofuran containing a trace Et3N treated at 0° with 0.5 cc. ClCO2Et, diluted after a few min. with absolute MeOH, and warmed to room temperature with stirring gave 1.7 g. Me ester of XXXIII, m. 53-6°, which was converted with concentrated NH4OH to the amide of XXXIII, m. 95-7° (from tetrahydrofuran-Et2O). The emulsion tests were carried out by dissolving the substance in 20 cc. H2O (employing generally the maximum concn), and mixing the solution in an Omnimixer with 5 cc. Nujol containing 0.2 g. cholesterol.

L10 ANSWER 16 OF 18 MEDLINE on STN

ACCESSION NUMBER: 93170958 MEDLINE
DOCUMENT NUMBER: PubMed ID: 8436445
TITLE: Synthesis and biological activity of [L-hydroxyproline]3-tuftsin analogue and its alpha- or beta-O-D-glucosylated derivatives.
AUTHOR: Biondi L; Filira F; Rocchi R; Tzehoval E; Fridkin M
CORPORATE SOURCE: C.N.R., Department of Organic Chemistry, University of Padova, Italy.
SOURCE: International journal of peptide and protein research, (1993 Jan) Vol. 41, No. 1, pp. 43-51.
Journal code: 0330420. ISSN: 0367-8377.
PUB. COUNTRY: Denmark
DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)
LANGUAGE: English
FILE SEGMENT: Priority Journals
ENTRY MONTH: 199303
ENTRY DATE: Entered STN: 2 Apr 1993
Last Updated on STN: 2 Apr 1993
Entered Medline: 22 Mar 1993

AB Syntheses are described of the Hyp3-tuftsin analogue and of its derivatives alpha- or beta-O-glycosylated at the side chain function of the hydroxyproline residue. The carbohydrate-free tetrapeptide was prepared by reacting Z-Thr-Lys(Z)-OH with H-Hyp-Arg(NO2)-OBzl by the **mixed anhydride** procedure. In the synthesis of the alpha-glycosylated analogue the O-glycosyl amino acid was incorporated by reacting Boc-(Glc alpha+beta)Hyp-OH with H-Arg(NO2)-OBzl through the same procedure. The alpha-**glucosylated** dipeptide was isolated from the diastereomeric mixture, selectively deblocked, and **acylated** with Z-Thr-Lys(Z)-OH by the **mixed anhydride** procedure. In the preparation of the beta-**glucosylated** analogue the BOP procedure was used for reacting Boc-[Glc(Ac)4 beta]Hyp-OH with H-Arg(NO)2-OBzl was well as for the final coupling to tetrapeptide. Removal of protecting groups from crude tetrapeptides was achieved by catalytic hydrogenation. Deacetylation of the sugar moiety of the beta-**glucosylated** tetrapeptide was achieved by treatment with sodium methoxide in methanol. The synthetic compounds were isolated by ion exchange chromatography, and characterized by elemental analysis, amino acid analysis, optical rotation and proton NMR. Their capacity to evoke the release of interleukin 1 from mouse peritoneal macrophages and to modulate immunogenic activity of antigen-fed cells was evaluated, in comparison with tuftsin and rigin. All of the analogues were found to possess tuftsin-like activity.

L10 ANSWER 17 OF 18 MEDLINE on STN
ACCESSION NUMBER: 91210013 MEDLINE
DOCUMENT NUMBER: PubMed ID: 2019473
TITLE: Synthesis of glycosylated tuftsins and tuftsin-containing IgG fragment undecapeptide.
AUTHOR: Biondi L; Filira F; Gobbo M; Scolaro B; Rocchi R
CORPORATE SOURCE: Department of Organic Chemistry, University of Padova, Italy.
SOURCE: International journal of peptide and protein research, (1991 Feb) Vol. 37, No. 2, pp. 112-21.
Journal code: 0330420. ISSN: 0367-8377.
PUB. COUNTRY: Denmark
DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)
LANGUAGE: English
FILE SEGMENT: Priority Journals
ENTRY MONTH: 199105
ENTRY DATE: Entered STN: 16 Jun 1991
Last Updated on STN: 16 Jun 1991
Entered Medline: 29 May 1991

AB Syntheses are described of two new tuftsin derivatives containing a 2-acetamido-2-deoxy-D-galactopyranosyl unit alpha- or beta-glycosidically linked to the threonine's hydroxy side chain function and of the glycosylated undecapeptide corresponding to the tuftsin region of the heavy chain of IgG (amino acid sequence 289-299). The glycosylated tuftsins were synthesized by the solution procedure. Fmoc-[Gal NAc(Ac)3 alpha]Thr-OH and Fmoc-[GalNAc(Ac)3 beta]Thr-OH were allowed to react with H-Lys(Z)-Pro-Arg(NO2)-OBzl by the **mixed anhydride** procedure and the resulting glycosylated tetrapeptides were fully deblocked by catalytic hydrogenation followed by treatment with potassium cyanide, purified by ion exchange chromatography and characterized by analytical HPLC, elemental and amino acid analyses, optical rotation, and proton NMR spectroscopy. Synthesis of the glycosylated undecapeptide was achieved by the continuous flow solid phase procedure on 4-hydroxymethylphenoxyacetyl-norleucyl derivatized Kieselguhr-supported resin. Fmoc-amino acid symmetrical anhydrides or pentafluorophenyl esters, in the presence of N-hydroxybenzotriazole, were used as the **acylating** agents. To mimic the native sequence of the tuftsin region at the Fc-domain of immunoglobulin G a 2-acetamido-2-deoxy-beta-D-**glucopyranosyl** unit was N-glycosidically linked to the amide side chain of Asn 297. The glycosylated asparagine residue was introduced as N2-fluorenylmethyloxycarbonyl-N4-(2-acetamido-3,4,6-tri-O-acetyl-2 -deoxy-beta-D - **glucopyranosyl**)-asparagine pentafluorophenyl ester. After cleavage from the resin the glycopeptide was deprotected, purified by ion exchange chromatography, and characterized by analytical HPLC, amino acid analysis, high voltage electrophoresis, and proton NMR. The conformational features of the glyco-undecapeptide were determined by circular dichroism measurements both in water and in 98% trifluoroethanol. Results of biological assays will be published elsewhere.

L10 ANSWER 18 OF 18 MEDLINE on STN
ACCESSION NUMBER: 87193557 MEDLINE
DOCUMENT NUMBER: PubMed ID: 3570665
TITLE: Synthesis of modified tuftsins containing monosaccharides or monosaccharide derivatives.
AUTHOR: Rocchi R; Biondi L; Filira F; Gobbo M; Dagan S; Fridkin M
SOURCE: International journal of peptide and protein research, (1987 Feb) Vol. 29, No. 2, pp. 250-61.
Journal code: 0330420. ISSN: 0367-8377.
PUB. COUNTRY: Denmark
DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)
LANGUAGE: English
FILE SEGMENT: Priority Journals
ENTRY MONTH: 198706

ENTRY DATE: Entered STN: 3 Mar 1990
Last Updated on STN: 3 Mar 1990
Entered Medline: 8 Jun 1987

AB Synthesis of some modified tuftsins is described in which a monosaccharide or a monosaccharide derivative was incorporated in the molecule. **Acylation** of H-Thr-Lys(Z)-Pro-Arg(NO2)-OBzl with D(+)-**gluco**-1,5-lactone followed by catalytic hydrogenation gave N alpha-**gluconyl**-tuftsin. Glycosylation of the carboxyl function of the C-terminal arginine has been achieved by reacting, through the **mixed anhydride** procedure, Boc-Thr-Lys(Z)-Pro-OH with 2-deoxy-2-(NG-nitroargininamido)-D-**glucopyranose** followed by catalytic hydrogenation and trifluoroacetic acid treatment. O-**Glucosyl**-tuftsin has been prepared by reacting o-nitrophenyl N-benzyloxycarbonyl-O-[(alpha + beta) 2,3,4,6-tetra-O-benzyl-D-**glucopyranosyl**]-threoninate with H-Lys(Z)-Pro-Arg(NO2)-OBzl in the presence of 1-hydroxybenzotriazole. Flash chromatography on silica gel allowed a partial separation of the diastereoisomers, one of which has been isolated in a reasonable yield. The single diastereoisomer and the alpha + beta anomeric mixture were separately deblocked by catalytic hydrogenation and purified by RP-HPLC.

L11 ANSWER 1 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1997:316151 CAPLUS
DOCUMENT NUMBER: 126:294804
TITLE: Studies on the selective degradation of chitin and the synthesis of the derivatives of glucosamine
AUTHOR(S): Lin, Yongcheng; Wang, Zhicai; Long, Zhongtao; Liao, Mingxiang; Wu, Xiongyu
CORPORATE SOURCE: Dep. Chem., Zhongshan Univ., Canton, 510275, Peop. Rep. China
SOURCE: Zhongguo Haiyang Yaowu (1996), 15(3), 1-4
CODEN: ZHYAE8; ISSN: 1002-3461
PUBLISHER: Shandong Haiyang Yaowu Kexue Yanjiuso
DOCUMENT TYPE: Journal
LANGUAGE: Chinese
AB The 4 compds. pentaacetylglucosamine, octaacetyldiglucosamine, undecaacetyltriglucosamine, and tetradecaacetyl tetraglucosamine were obtained by hydrolysis and acetolysis of chitin. Using **glucosamine** as raw material, 6 **glucosamine** derivs. were synthesized by **mixed anhydride** or DCC methods: N-(N'-Boc-1-prolyl)-O-tetraacetylglucosamine, N-(N'-phthaloyl-L-leucyl)-O-tetraacetyl-D-**glucosamine**, N-(n'-phthaloyl-L-valyl)-O-tetraacetylD-**glucosamine**, N-(N'-phthaloyl-L-phenylalanyl)-O-tetraacetyl-D-**glucosamine**, N-benzoyl-O-tetraacetyl-D-**glucosamine** and N-**acetylsalicyl**-O-tetraacetyl-D-**glucosamine**.

L11 ANSWER 2 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1996:731998 CAPLUS
DOCUMENT NUMBER: 126:8722
TITLE: Preparation of asparagine-containing glycopeptides
INVENTOR(S): Inazu, Toshuki
PATENT ASSIGNEE(S): Noguchi Kenkyusho, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 08245688	A2	19960924	JP 1995-80731	19950313

PRIORITY APPLN. INFO.: JP 1995-80731 19950313
OTHER SOURCE(S): CASREACT 126:8722
AB Asparagine-containing glycopeptides are prepared by using a Nα-protected-Nβ-glycosyl-L-asparagine thiophosphinic acid **mixed anhydride** as a synthetic unit. The Nα-amino protecting group is preferably 9-fluorenylmethoxycarbonyl group and the thiophosphinic **mixed anhydride** is the dimethylthiophosphinic acid **mixed anhydride**. Hydroxy group-unprotected derivative of a sugar is also used. The peptide coupling reaction is also carried out on a solid-phase support. This synthetic unit allows the use of hydroxy group-unprotected sugar derivs., which eliminates the deprotection of the hydroxy protecting groups at the final stage of the peptide synthesis. The glycopeptides are useful as drugs, agrochems., and food additives and as ligands for studying cell recognition mechanism (no data). Thus, 100 mg Nα-(9-fluorenylmethoxycarbonyl)-[Nβ-(2-acetamido-3,4,6-tri-O- **acetyl** -2-deoxy-β-D- **glucopyranosyl**)]-L-asparagine was dissolved in DMF, treated with (Me2CH)2NEt and dimethylphosphinothioyl chloride, and stirred at room temperature for 30 min to prepare the corresponding dimethylthiophosphinic acid **mixed anhydride**, which was condensed with Et glycinate hydrochloride in the presence of (Me2CH)2NEt

in DMF to give 80% Nα-(9-fluorenylmethoxycarbonyl)-[Nβ-(2-acetamido-3,4,6-tri-O-**acetyl**-2-deoxy-β-D-**glucopyranosyl**)]-L-asparaginylglycine Me ester.

L11 ANSWER 3 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1996:253654 CAPLUS
DOCUMENT NUMBER: 124:307808
TITLE: Conjugates of insulin with copolymers of N-(2-hydroxypropyl) methacrylamide: effects on smooth muscle cell proliferation
AUTHOR(S): Chytry, Vladimir; Letourneur, Didier; Baudys, Miroslav; Jozefonvicz, Jacqueline
CORPORATE SOURCE: Inst. Macromolecular Chemistry, Academy Sciences Czech Republic, Prague, Czech Rep.
SOURCE: Journal of Biomedical Materials Research (1996), 31(2), 265-72
CODEN: JBMRBG; ISSN: 0021-9304
PUBLISHER: Wiley
DOCUMENT TYPE: Journal
LANGUAGE: English
AB The hypothesis that an elevated plasma insulin level contributes to an increase in coronary heart disease has led to studies of the mitogenic effect of native insulin and its conjugates on smooth muscle cells (SMC). In this study, insulin was covalently attached to two water-soluble polymers containing N-(2-hydroxypropyl)methacrylamide using the **mixed anhydride** method. The first polymer was a copolymer of N-(2-hydroxypropyl)methacrylamide and N-methacryloyldiglycine. The second one was a terpolymer of two of the above-given monomers and R-(-)-1-methyl-2-methacryloylamidoethyl 2-acetamido-2-deoxy-β-D-**glucopyranoside**. Insulin conjugates were isolated and characterized, and the mitogenic effect on SMC was investigated. The results showed that only conjugates of insulin and terpolymers bearing pendant N-**acetyl**-**glucosamine** groups do not have a mitogenic effect on SMC while maintaining the hypoglycemic activity of insulin. This finding suggests that some inter- or intramol. interactions of coupled insulin with the sugar moiety(ies) attached to the polymer backbone contribute to the observed effects.

L11 ANSWER 4 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1992:592210 CAPLUS
DOCUMENT NUMBER: 117:192210
TITLE: Syntheses of N-acyl and N-alkoxycarbonyl derivatives of 2-[(alkoxycarbonyl)amino]-2-deoxy-D-glucose.
AUTHOR(S): Lafont, Dominique; Boullanger, Paul
CORPORATE SOURCE: ESCIL, Univ. Lyon I, Villeurbanne, 69622, Fr.
SOURCE: Journal of Carbohydrate Chemistry (1992), 11(5), 567-86
CODEN: JCACDM; ISSN: 0732-8303
DOCUMENT TYPE: Journal
LANGUAGE: English
AB N-Acyl and N-alkoxycarbonyl derivs. of 1,3,4,6-tetra-O-**acetyl**-2-alkoxycarbonylamino-2-deoxy-β-D-**glucopyranose** were synthesized using **mixed anhydrides** and sym. or unsym. pyrocarbonates. These derivs. were used as donors in 1,2-trans-glycosylation reactions promoted by Lewis acids. Besides the expected β-D-glycosides, cyclization and rearrangement side-products were often encountered in such glycosylations.

L11 ANSWER 5 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1991:81345 CAPLUS
DOCUMENT NUMBER: 114:81345
TITLE: Chiral synthesis of polyketide-derived natural products. 31. Stereoselective synthesis of erythronolide A by extremely efficient lactonization

based on conformational adjustment and high activation of seco-acid

AUTHOR(S): Hikota, Masataka; Tone, Hitoshi; Horita, Kiyoshi; Yonemitsu, Osamu

CORPORATE SOURCE: Fac. Pharm. Sci., Hokkaido Univ., Sapporo, 060, Japan

SOURCE: Tetrahedron (1990), 46(13-14), 4613-28
CODEN: TETRAB; ISSN: 0040-4020

DOCUMENT TYPE: Journal

LANGUAGE: English

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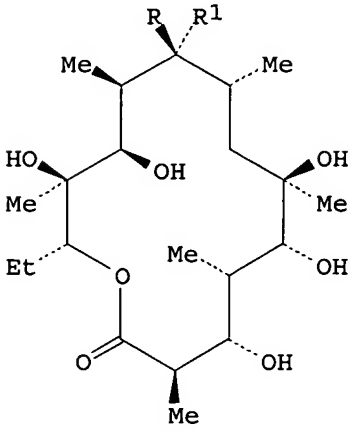

I

AB C(1)-C(5) sulfone and C(7)-C(15) aldehyde, synthesized stereoselectively from D-**glucose**, were coupled, and the C(5) and C(6) chiral centers were constructed taking advantage of the 4-methoxybenzyl-type protecting group to give the protected seco acid. When the **mixed anhydride** of the seco acid was treated with a high concentration of 4-dimethylaminoypridine at room temperature, a very rapid cyclization occurred and the 14-membered lactone was isolated in almost quant. yield. Deprotection readily gave 9-dihydroerythronolide A (I, R = OH, R1 = H), which was converted to I (RR1 = O).

L11 ANSWER 6 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1989:595384 CAPLUS

DOCUMENT NUMBER: 111:195384

TITLE: Synthesis and reactions of O-**acetylated** benzyl α-glycosides of 6-O-(2-acetamido-2-deoxy-β-D-glucopyranosyl)-N- **acetylmuramoyl** -L-alanyl-D-isoglutamine esters: the base-catalyzed isoglutamine .dblarw. glutamine rearrangement in peptidoglycan-related structures

AUTHOR(S): Keglevic, Dina; Derome, Andrew E.

CORPORATE SOURCE: Dep. Org. Chem. Biochem., "Ruder Boskovic" Inst., Zagreb, 41001, Yugoslavia

SOURCE: Carbohydrate Research (1989), 186(1), 63-75
CODEN: CRBRAT; ISSN: 0008-6215

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 111:195384

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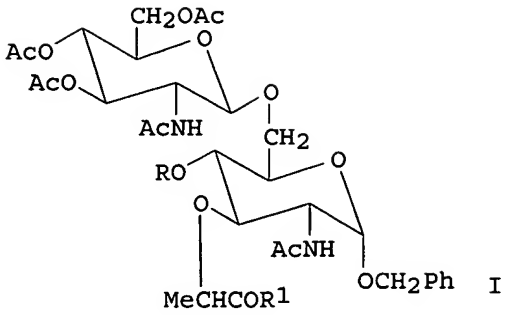

I

AB Condensation of **glucopyranosides** I (R = H, Ac, R1 = OH) with H-Ala-D-Glu(OCH2Ph)-NH2 via the **mixed anhydride** method yielded peptidoglycans I [R = H, Ac, R1 = Ala-D-Glu(OCH2Ph)-NH2] (II). O-Deacetylation of II with NaOMe in MeOH led to transesterification and α→γ transamidation of the isoglutaminyl residue to give isoglutamine and glutamine Me esters. Treatment of II with MgO-MeOH caused deacetylation at the GlcNAc residue to give a mixture of isoglutamine and glutamine Me esters. Thus, benzyl or Me ester protection of peptidoglycan-related structures is not compatible with any reactions requiring alkaline media. Condensation of I (R = H, R1 = OH) with H-Ala-D-Glu(OCMe3)-NH2 gave peptidoglycan I [R = H, R1 = Ala-D-Glu(OCMe3)-NH2], deacetylation of which, with NaOMe, proceeded without side reactions to afford N-[2-O-[benzyl 2-acetamido-6-O-(2-acetamido-2-deoxy-β-D- **glucopyranosyl**)-2,3-dideoxy-α-D- **glucopyranosid**-3-yl]-(R)-lactoyl]-L-alanyl-D-isoglutamine tert-Bu ester.

L11 ANSWER 7 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1988:510849 CAPLUS
DOCUMENT NUMBER: 109:110849
TITLE: Preparation and testing of glycosylamides as immunostimulants
INVENTOR(S): Lockhoff, Oswald; Hayauchi, Yutaka; Stadler, Peter; Brunner, Helmut
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.
SOURCE: Ger. Offen., 17 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3604681	A1	19870820	DE 1986-3604681	19860214
US 4891425	A	19900102	US 1987-7703	19870128
EP 234348	A2	19870902	EP 1987-101462	19870203
EP 234348	A3	19890426		
EP 234348	B1	19910529		
R: AT, BE, CH, DE, ES, FR, GB, IT, LI, NL, SE				
AT 63920	E	19910615	AT 1987-101462	19870203
ES 2039211	T3	19930916	ES 1987-101462	19870203
CA 1271190	A1	19900703	CA 1987-529610	19870212
JP 62192397	A2	19870822	JP 1987-29921	19870213

PRIORITY APPLN. INFO.: DE 1986-3604681 A 19860214
EP 1987-101462 A 19870203

OTHER SOURCE(S): MARPAT 109:110849
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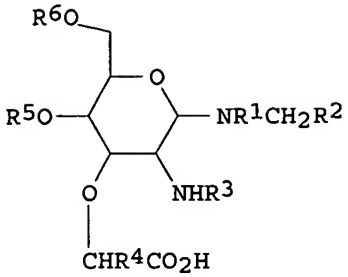
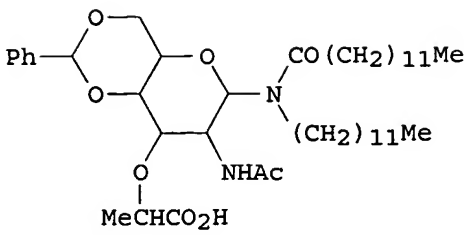


I



II

AB The title compds. [I; R1, R2 = H, (un)saturated C1-50 alkyl; R3 = H, acyl; R4 = H, alkyl; R5, R6 = H, acyl, alkyl, aralkyl; R5R6 = R7R8C; R7,R8 = H, alkyl, (substituted) aryl] were prepared as immunostimulants. 2-**Acetylamino**-2-desoxy-D-**glucose** was aminated with Me(CH2)11NH2, acylated with the **mixed anhydride** from Me(CH2)11CO2H and EtO2CCl, and ketalized with PhCH(OMe)2/tosic acid. The product was deprotonated with NaH in dioxane at 95°, and L-2-chloropropanoic acid was added at 60° to give amino sugar II. II at 10 mg/kg orally in rats reduced the number of Salmonella typhimurium in the blood to 4% of controls.

L11 ANSWER 8 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1988:493618 CAPLUS
DOCUMENT NUMBER: 109:93618
TITLE: Synthesis of glycosylated enkephalin analogs with potent opiate activity
INVENTOR(S): Garcia Anton, J. Maria; Torres, J. L.; Rodriguez, R.; Reig, F.
PATENT ASSIGNEE(S): Consejo Superior de Investigaciones Cientificas, Spain
SOURCE: Span., 11 pp.
CODEN: SPXXAD
DOCUMENT TYPE: Patent
LANGUAGE: Spanish
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ES 549220	A1	19870501	ES 1985-549220	19851122

PRIORITY APPLN. INFO.: ES 1985-549220 19851122

AB Enkephalin analogs H-Tyr-D-Met-Gly-Phe-Pro-X [I; X = D-**glucopyranosylamino**, 2-deoxy-D-**glucopyranosyl**-2-amino, 2-deoxy-6-sulfo-D-**glucopyranosyl**-2-amino], useful as analgesics (no data), are prepared in 5 steps. Coupling of Z-Gly-OR (Z = PhCH2OCO, R = succinimido) with phenylalanine in aqueous dioxane containing NaHCO3 gave 86% Z-Gly-Phe-OH, which was hydrogenolyzed (82%) and esterified by SOCl2/MeOH (81.7%) to give H-Gly-Phe-OMe. Coupling of the dipeptide with Boc-D-Met-OH (Boc = tert-BuOCO) via the **mixed anhydride** from iso-BuOCOCl gave 98% tripeptide, which was N-deprotected by CF3CO2H (92%), coupled with Boc-Tyr-OH using DCC and hydroxybenzotriazole (70%), and saponified by 1 N NaOH in Me2CO (90%) to give Boc-Tyr-D-Met-Gly-Phe-OH. Coupling of the tetrapeptide with H-Pro-OMe·HCl by the DCC method (73.9%), followed by saponification as above (96%), DCC-mediated coupling with tetra-O-**acetyl**-D-**glucopyranosylamine**, and sequential deprotection with CF3CO2H and NH3-saturated MeOH gave I (X = D-**glucopyranosylamino**).

L11 ANSWER 9 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1981:604347 CAPLUS
DOCUMENT NUMBER: 95:204347
TITLE: Amino sugars. 127. The synthesis of uridine

diphosphate N-**acetylhexosamines** and uridine 5'-(2-acetamido-2-deoxy-α-D-mannopyranosyluronic acid diphosphate)

AUTHOR(S): Yamazaki, Tatsumi; Warren, Christopher D.; Herscovics, Annette; Jeanloz, Roger W.

CORPORATE SOURCE: Dep. Biol. Chem., Harvard Med. Sch., Boston, MA, 02114, USA

SOURCE: Canadian Journal of Chemistry (1981), 59(15), 2247-52
CODEN: CJCHAG; ISSN: 0008-4042

DOCUMENT TYPE: Journal

LANGUAGE: English

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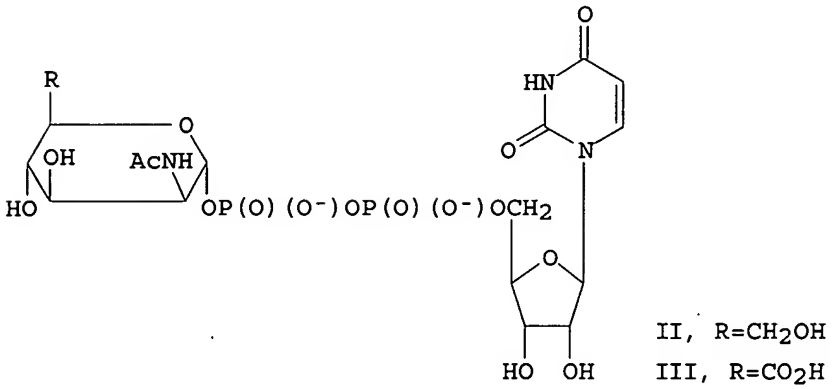


II, R=CH_2OH

III, R=CO_2H

AB 2-Methyl-(2-acetamido-3,4,6-trio-O-**acetyl**-1,2-dideoxy-β-D-mannopyrano)-[2,1-d]-2-oxazoline was efficiently converted into 2-acetamido-3,4,6-tri-O-**acetyl**-2-deoxy-α-D-mannopyranosyl phosphate (I), by treatment with dibenzyl phosphate, followed by catalytic hydrogenolysis of the benzyl groups. Similarly, 2-acetamido-3,4,6-tri-O-**acetyl**-2-deoxy-α-D- **glucopyranosyl** phosphate and -galactopyranosyl phosphate were synthesized from the resp. peracetyl oxazolines. In each case, the procedures for preparing the oxazoline, and conversion into the glycosyl phosphate, were modified to give high yields of pure products. I was coupled with 2',3'-di-O-**acetyluridine** 5'-monophosphate by a modification of the **mixed anhydride** procedure, to give 2',3'-di-O-**acetyluridine** 5'-(2-acetamido-3,4,6-tri-O-**acetyl**-2-deoxy-α-D-mannopyranosyl diphosphate), which was readily purified by preparative TLC and O-deacetylated to give uridine diphosphate N-**acetylmannosamine** (II) in high yield. Similarly, uridine 5'-(2-acetamido-2-deoxy-α-D-**glucopyranosyl**- and -galactopyranosyl diphosphates) were synthesized by rapid, efficient procedures, not involving ion-exchange chromatog. II was converted into mannopyranosyluronic acid III, required for biosynthetic studies, without the preparation of a special Pt catalyst. All the synthetic uridine diphosphate sugars were characterized by optical rotation, 1H NMR spectrum, and elemental anal.

L11 ANSWER 10 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1980:181640 CAPLUS

DOCUMENT NUMBER: 92:181640

TITLE: Bacterial cell wall constituents, Part III. Synthesis of O-(2-acetamido-2-deoxy-β-D-glucosyl)-(1 → 4)-N-(**acetylmuramoyl**)-L-alanyl-D-isoglutamine, the repeating disaccharide-dipeptide unit of the bacterial cell wall

peptidoglycan
AUTHOR(S): Durette, Philippe L.; Meitzner, Eric P.; Shen, T. Y.
CORPORATE SOURCE: Merck Sharp and Dohme Res. Lab., Rahway, NJ, 07065, USA
SOURCE: Carbohydrate Research (1979), 77, C1-C4
CODEN: CRBRAT; ISSN: 0008-6215
DOCUMENT TYPE: Journal
LANGUAGE: English
GI For diagram(s), see printed CA Issue.
AB The title glycopeptide (I) was prepared by alkylating disaccharide II (R = CH2Ph, R1 = NHAc, R3 = H) (III) by (S)-MeCHClCO2H, condensing the resulting muramic acid IV (R3 = OH) with H-Ala-D-Glu(OCH2Ph)-NH2 by the **mixed anhydride** method, and deblocking the resulting IV [R3 = Ala-D-Glu(OCH2Ph)-NH2] by hydrogenolysis. Glycopyranosyl chloride V (PhtN = phthalimido) was treated with **glucopyranoside** VI to give II (R = Ac, R1 = PhtN, R2 = allyl), which was deacetylated-dephthaloylated and then N-**acetylated** with Ac2O to give II (R = H, R1 = NHAc, R2 = allyl). The latter was benzylated to give II (R = CH2Ph, R1 = NHAc, R2 = allyl), which was deallylated to give III.

L11 ANSWER 11 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1977:547477 CAPLUS
DOCUMENT NUMBER: 87:147477
TITLE: The preparation of carbohydrate-protein conjugates: cyanuric trichloride coupling of 2-aminoethyl glycosides, and mixed-anhydride coupling of 8-carboxyoctyl glycosides to bovine serum albumin
AUTHOR(S): King, Russell R.; Cooper, Fred P.; Bishop, Claude T.
CORPORATE SOURCE: Div. Biol. Sci., Natl. Res. Counc. Canada, Ottawa, ON, Can.
SOURCE: Carbohydrate Research (1977), 55, 83-93
CODEN: CRBRAT; ISSN: 0008-6215
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Preparation of the following glycosides is described: 2-aminoethyl β-D-glycosides of (A) 2-acetamido-3,4,6-tri-O- **acetyl** -2-deoxy-D-**glucopyranose**, (B) 2-acetamido-4-O-(2-acetamido-3,4,6-tri-O-**acetyl**-2-deoxy-β-D- **glucopyranosyl**)-3,6-di-O-**acetyl**-2-deoxy-β-D- **glucopyranose** (N,N'-diacetylchitobiose pentaacetate), (C) 4-O-(2,3,4,6-tetra-O-**acetyl**-β-D- **glucopyranosyl**)-2,3,6-tri-O-**acetyl**-β-D- **glucopyranose** (cellobiose heptaacetate); 8-carboxyoctyl glycosides of (D) cellobiose, and (E) N,N'-diacetylchitobiose. Conjugates were prepared from (A), (B), and (C) by coupling to bovine serum albumin by cyanuric trichloride and subsequent deacetylation; (D) and (E) were coupled to bovine serum albumin by the **mixed anhydride** reaction. Conjugates (A) and (B) were insol.; conjugates (C), (D), and (E) functioned as artificial antigens and gave rise to precipitating antibodies in rabbits. Specificities of the antisera were determined by inhibition studies.

L11 ANSWER 12 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1968:496329 CAPLUS
DOCUMENT NUMBER: 69:96329
TITLE: Synthesis and characterization of compounds related to daunomycin
AUTHOR(S): Marsh, John P., Jr.; Iwamoto, Robert H.; Goodman, Leon
CORPORATE SOURCE: Stanford Res. Inst., Menlo Park, CA, USA
SOURCE: Chemical Communications (London) (1968), (10), 589-90
CODEN: CCOMA8; ISSN: 0009-241X
DOCUMENT TYPE: Journal
LANGUAGE: English
GI For diagram(s), see printed CA Issue.

AB Condensation of 5,8-dimethoxy-1,2,3,4-tetrahydronaphthalene with the **mixed anhydride** of Me hydrogen phthalate and CF3CO2H gave 6-[2-(methoxycarbonyl)benzoyl] - 5,8-dimethoxy - 1,2,3,4-tetrahydronaphthalene which was saponified to 6-(2-carboxybenzoyl)-5,8-dimethoxy-1,2,3,4-tetrahydronaphthalene (I). I was subjected to ring closure with H2SO4 and demethylated with AlCl3 to II (R = H), which was brominated with Me4NBr3 to a mixture of II (R = Br), some dibromide, and unchanged II (R = H). This bromination mixture was treated with AgOAc to give a mixture of II (R = H), II (R = OAc), and some diacetate. II (R = OAc) dissolved in CF3CO2H was converted to II (R = O2CCF3) which was methanolyzed to II (R = OH), m. 292-4°. II (R = OH) reacted with tetra-O-**acetyl**-α-D- **glucopyranosyl** bromide in the presence of Hg(CN)2 to give III (R = Ac) as a mixture of diastereoisomers, m. 235-42°, which was treated with methanolic NaOMe to give III (R = H), m. >300°. Preparative thin layer chromatog. of III (R = Ac) on silicic acid gave a faster-moving **glucoside** (IV), m. 235°, [α]D 106° (dioxane), and a slower-moving component, m. 268°, [α]D -216° (dioxane). IV has the same configuration at the benzylic C as daunomycin and daunomycinone, as indicated by circular dichroism spectra.

L11 ANSWER 13 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1964:83166 CAPLUS
DOCUMENT NUMBER: 60:83166
ORIGINAL REFERENCE NO.: 60:14603b-g
TITLE: Synthesis of peptides of 2-amino-2-deoxy-D-glucose as a contribution to the question of the linkage of carbohydrate to protein in glycoproteins and mucopolysaccharide proteins
AUTHOR(S): Wacker, Oskar; Lieflaender, Manfred
CORPORATE SOURCE: Max-Planck-Ges., Goettingen, Germany
SOURCE: Hoppe-Seyler's Zeitschrift fuer Physiologische Chemie (1964), 335(2), 255-71
CODEN: HSZPAZ; ISSN: 0018-4888
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB Peptides of 2-amino-2-deoxy-D-**glucose** (I) were synthesized by 3 methods: (1) reaction of N-carbobenzoxy (Cbo) peptides with 1,3,4,6-O-tetraacetyl-2-amino-2-deoxy-β-D- **glucose** (II) by the **mixed anhydride** method with ClCO2Et; (2) reaction of N-Cbo-peptides or N-formyl peptides with an N-aminoacyl derivative of II by the **mixed anhydride** method; and (3) **mixed anhydride** synthesis from N-Cbo-peptides and I. N-(N-Cbo-Gly-Gly) derivative of II [[α]27D 13.7° (c 1.2, CHCl3), m. 151-3°], obtained in 44% yield by method 1, was deacetylated with NaOMe to give 50% N-(N-Cbo-Gly-Gly) derivative of I [[α]27D 50° (c 1.3, MeOH), m. 187-90°]. N-L-Valyl derivative of II acetate [[α]27D -10.4° (c 1.3, CHCl3), decompose on heating, 81% yield], N-L-leucyl derivative of II acetate [[α]25D 12.4° (c 2, MeOH), m. 148-50°, 82% yield], N-L-lysyl derivative of II diacetate [[α]25D 18° (c 2.1, MeOH), m. 148-52°, 54% yield], N-L-tyrosyl derivative of II [[α]25D 29.7° (c 0.6, MeOH), m. 198-200°, 69% yield], and N-L-glutamyl γ-Et ester derivative of II acetate [[α]25D 16.3° (c 2.2, MeOH), m. 119-21°, 69% yield] were prepared from the corresponding N-Cbo-amino acids and II by the **mixed anhydride** method, followed by catalytic hydrogenation. By method 2, N-L-leucyl derivative of II was treated with N-Cbo-L-leucine and N-Cbo-L-Leu-L-Leu to give, resp., 65% N-(N-Cbo-L-Leu-LLeu) derivative of II [[α]26D 14.1° (c 0.7, MeOH), m. 229-30°], and 39% N-(N-Cbo-L-Leu-L-Leu-L-Leu) derivative of II [[α]26D 15.8° (c 1.3, MeOH), m. 228°]. Similarly, N-L-valyl derivative of II treated with N,N'-di-Cbo-L-lysine, N,O-di-Cbo-L-tyrosine, and N-formyl-DL-methionine gave, resp., 63% N-(N,N'di-Cbo-L-Lys-L-Val) derivative of II [[α]25D -8.8° (c

1.1., CHCl3), m. 205-7°], 51% N-(N,O-di-Cbo-L-Tyr-L-Val) derivative of II [[α]25D 9.2° (c 1, CHCl3), m. 234-6°], and 66% N-(N-formyl-DL-Met-L-Val) derivative of II [[α]27D -21.8° (c 1.2, CHCl3), m. 211-14°]. Deacetylation of the above 5 derivs. of II with NaOMe yielded, resp., 48% N-(N-Cbo-L-Leu-L-Leu) derivative of I [[α]25D -4.9° (c 2, MeOH), m. 200-2°], 47% N-(N-Cbo-L-Leu-L-Leu-L-Leu) derivative of I [[α]25D -10.1° (c 0.5, MeOH), m. 202-4°], 59% N-(N,N'-di-Cbo-L-Lys-L-Val) derivative of I [[α]25D 35° (c 0.9, Me2NCHO), m. 201-3°], 56% N-(N-Cbo-L-Tyr-L-Val) derivative of I [α]25D 58.8° (c 0.8, Me2NCHO), m. 222-6°], and 82% N-(N-formyl-DL-Met-L-Val) derivative of I, [α]25D 96.6° (c 0.6, Me2NCHO), m. 218-20°. Catalytic hydrogenation of the Cbo-compds., and hydrolysis with HCl of the formyl compound gave the following corresponding free peptide hydrochlorides in 72-86% yields: N-(L-Leu-L-Leu) derivative of I-HCl [[α]25D 19.3° (c 1.9, H2O)]; N-(L-Leu-L-Leu-L-Leu) derivative of I-HCl [[α]25D -7.5° (c 0.7, H2O)]; N-(L-Lys-L-Val) derivative of I-HCl [[α]25D 25.8° (c 0.8, H2O)]; N-(L-Tyr-L-Val) derivative of I-HCl [[α]25D 26.6° (c 0.9, H2O)]; and N-(DL-Met-L-Val) derivative of I-HCl, [α]25D 22.9° (c 0.3, H2O). N-(N-Cbo-L-Leu-L-Leu) derivative of I and N-(N,N'-di-Cbo-L-Lys-L-Val) derivative of I were also prepared directly from the substituted dipeptides and I by method 3, in yields of 67 and 46%, resp. When peptide hydrochlorides of I were treated with alkali, 4-8 substances giving colored products with p-dimethylaminobenzaldehyde resulted. The Elson-Morgan reaction on pepdides of I gave approx. 50% of the color values of the N-**acetyl** derivative of I.

L11 ANSWER 14 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1963:66765 CAPLUS
DOCUMENT NUMBER: 58:66765
ORIGINAL REFERENCE NO.: 58:11462h,11463a-d
TITLE: Synthesis of N-(β-L-asparagyl)-D-glucosamine, O-β-methyl-N-(α-mehtyl-β-L-asparagyl)-D-glucosaminide, and their derivative
AUTHOR(S): Stakheeva-Kaverzneva, E. D.; Konovalova, M. I.
CORPORATE SOURCE: N. D. Zelinskii Inst. Org. Chem., Moscow
SOURCE: Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1963) 124-8
CODEN: IASKA6; ISSN: 0002-3353
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 58:66765
GI For diagram(s), see printed CA Issue.
AB L-Carbobenzoxyaspartic anhydride and PhCH2OH in 4 hrs. at 100° in an ampul gave L-carbobenzoxyaspartic acid α-benzyl ester, m. 85-5.2° (Bergmann, et al., CA 27, 5723), which, treated with EtO2CCl in Et3N-tetrahydrofuran at -10° followed after 15 min. by 1,3,4,6-tetra-O-**acetyl**-β-D-**glucosamine**-HCl (decomposed 225-7°, [α]23D 28.5°) gave after 15 min. at 0° and 3-4 hrs. at 20° N-(α-benzyl-N'-carbobenzoxy-β-asparagyl)-1,3,4,6-tetra-O-**acetyl**-β-D-**glucosamine** (I), 75.2%, m. 205° (EtOH), m. 206-6.7° (from Me2CO), [α]23D 11° (Marks and Neuberger, CA 56, 13006c). This and MeONa in CHCl3 at -10° 10 min. and at 20° 10 min. gave 67.7% N-(α-methyl-N'-carbobenzoxy-β-asparagyl)-D-**glucosamine**, decomposed 160-1°, [α]22D 25.3°. The solution of the **mixed anhydride** formed from α-benzyl carbobenzoxy-L-aspartate and EtO2CCl, as above in tetrahydrofuran, treated at 0° with D-**glucosamine** in tetrahydrofuran-MeOH and after 0.5 hr. at -10° and overnight at 20°, gave 50% N-(α-benzyl-N'-carbobenzoxy-β-asparagyl)-D-**glucosamine**, m. 167-8°, [α]23D 20°, which

was hydrogenated over Pd in aqueous MeOH-Me2CO to N-(L-β-asparagyl)-D-**glucosamine** (II), 36%, m. above 320° [α]19D 30.2°. I kept 20 hrs. at 0° with Ba(OMe)2 in MeOH, then treated with CO2, filtered, evaporated in vacuo, and hydrogenated over Pd directly gave 59% II; pure II had mutarotating behavior with [α]23D changing in H2O from 31.9° to 22.7° the substance was evidently a monohydrate. Refluxing I 2 hrs. with 2% HCl-MeOH, followed by shaking with PbCO3 (operation repeated) gave O-β-methyl-N-(α-methyl-N'-carbobenzoxy-β-asparagyl)-D- **glucosaminide** (III), amorphous solid (MeOH-Et2O), [α]23D 63°. This does not react with alkaline Ag solns. Hydrogenation in MeOH in the presence of AcOH over Pd gave 81% O-methyl-N-(α-methyl-β-asparagyl)-D-**glucosaminide** hemihydrate acetate, a slowly solidifying oil, [α]23D 80.6°.

L11 ANSWER 15 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1961:111760 CAPLUS
DOCUMENT NUMBER: 55:111760
ORIGINAL REFERENCE NO.: 55:20977c-i,20978a
TITLE: β-D-Glucosylamides of L-amino acids and of nicotinic acid
AUTHOR(S): Coutsogeorgopoulos, Charalambos; Zervas, Leonidas
CORPORATE SOURCE: Univ. Athens, Greece
SOURCE: Journal of the American Chemical Society (1961), 83, 1885-8
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 55:111760

AB 4,6-O-Benzylidene-D-**glucose** (20.0 g.) was added to 35-40 g. NH3 in 120 ml. MeOH at -20° in a steel bomb, the mixture shaken 3 hrs. at 60°, cooled to 0-5°, concentrated to remove NH3, the product filtered off, and washed with MeOH at 4° to give 46% 4,6-O-benzylidene-β-D- **glucosylamine** (I), m. 166-8° (decomposition) (MeOH or dioxane), [α]18D -67.2° (c 2, C5H5N, 8 min. after solution), -58° (after 48 hrs.); N-Ac derivative (82%) m. 242-3° (decomposition), [α]25D -41.7° (c 2, C5H5N). Catalytic hydrogenation with Pd black in 50% EtOH containing HOAc gave 75% known N-**acetyl**-β-D- **glucosylamine**. N-Benzoyl-4,6-O-benzylidene-β-D- **glucosylamine** (43%) m. 265-8° (decomposition) (EtOH), [α]15D -65.8° (c 2, C5H5N); this was similarly converted to 75% known N-benzoyl-β-D-**glucosylamine**, m. 238-42°, [α]17D -13.8° (c 2, H2O). Reaction of 3 g. carbobenzoxy-L-phenylalanine, 1.4 ml. Et3N, and 0.97 ml. ClCO2Et in 25 ml. dioxane for 10 min. at 10°, addition with shaking to 2.7 g. I in 80 ml. dioxane, the mixture shaken 4 hrs. at 20°, removal of solvent and crystallization from EtOH gave 60% N-(N-carbobenzoxy-L-phenylalanyl)-4,6-O-benzylidene-β-D-**glucosylamine**, m. 245-7° (decomposition), [α]20D -25.6° (c 2, C5H5N); this product catalytically hydrogenated until CO2 evolution ceased, the product heated in 0.1N HCl at 100° for 30 min., concentrated to dryness, reconcd. from EtOH solution, and the residue treated in EtOH with Et3N and chilled gave 77% N-(L-phenylalanyl)-β-D-**glucosylamine**, sintered at 115-20°, melted over a higher range, [α]20D -2.6° (c 9, 0.3N HCl). Similarly, N-carbobenzoxy-α-benzyl-L-aspartate and I gave 66% benzyl Nα-carbobenzoxy-Nγ-(4,6-O-benzylidene-β-D-**glucosyl**)-L-asparaginate, m.200-3°, [α]20D -21.3° (c 2, C5H5N), converted to 86% N-(L-β-aspartyl)-β-D-**glucosylamine**, m. 253° (decomposition), [α]16D -16.5° (c 2, H2O). α-Benzyl-N-carbobenzoxy-L-glutamate (II) and I gave 64% benzyl N-carbobenzoxy-N-(4,6-O-benzylidene-β-D-**glucosyl**)-L-glutaminate, m. 192-3°, [α]16D

-11.0° (c 2, dioxane), converted to 79% N-(L-γ-glutamyl)-
β-D- **glucosylamine**, m. 211°, [α]17D
-9.7° (c 2, H2O); the tetrahydrate also m. 211° (EtOH-H2O).
Nicotinic acid and I similarly gave 38% N-nicotinoyl-4,6-O-benzylidene-
β-D- **glucosylamine** (III), m. 250-2°, [α]15D
-60.0° (c 0.5, C5H5N); adding 1.8 g. fresh nicotinoyl chloride HCl
salt during 15 min. to 2.7 g. I in 25 ml. C5H5N, shaking the mixture 30
min., and adding to ice H2O also gave 32% III. Heating 2.25 g. III in 60
ml. 0.1N HCl at 100° for 20 min., concentrating the mixture to dryness,
treating the residue with 0.8 ml. Et3N in 3 ml. EtOH, and cooling gave 82%
N-(nicotinoyl)-β-D- **glucosylamine**, m. 234-5°
(decomposition), [α]15D -8.3° (c 3, H2O). α-Benzyl
L-glutamate (IV), used in preparation of II, was prepared as follows. Adding
5.6
g. Ph3CCl with cooling to 9.7 g. dibenzyl L-glutamate benzenesulfonate in
60 ml. CHCl3 and 6 ml. Et3N, letting the mixture stand overnight,
concentrating to
dryness and crystallizing from MeOH gave 90% dibenzyl N-trityl-L-glutamate (V);
treating 11.4 g. in 25 ml. Me2CO with 5.5 ml. 4N LiOH, alternately heating
to dissolve the precipitated LiOH and shaking at 20° during 4 hrs. until
solution was attained, diluting to 500 ml. with H2O, washing with Et2O,
acidifying with 25 ml. HOAc, and cooling to 4° gave 90%
α-benzyl N-trityl-L-glutamate, m. 105-15° (decomposition, with
sintering at 58-60°), [α]15D 40.3° (c 2, EtOH). This
was converted with Et3N and ClCO2Et to the **mixed**
anhydride, and then with dioxane-NH3 to 84% benzyl
Nα-trityl-L-glutaminate, m. 119-21°, [α]15D
49.7° (c 2, EtOH). Hydrogenation in MeOH, and crystallization from
EtOH-H2O gave 70% L-glutamine. Heating 4.8 g. V in 5 ml. HOAc and 0.5 ml.
H2O to the b.p., cooling the mixture, adding Me2CO to dissolve Ph3COH,
adding Et2O, and cooling gave 82% IV.

L11 ANSWER 16 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1956:52547 CAPLUS
DOCUMENT NUMBER: 50:52547
ORIGINAL REFERENCE NO.: 50:10048b-i,10049a
TITLE: Glycosidic constituent of Vinca minor and its identification as 3-β-D-glucosyloxy-2-hydroxybenzoic acid
AUTHOR(S): King, F. E.; Gilks, J. H.; Partridge, M. W.
CORPORATE SOURCE: Univ. Nottingham, UK
SOURCE: Journal of the Chemical Society (1955) 4206-15
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 50:52547
AB Finely powdered leaves and stems of V. minor extracted with 90% aqueous MeOH
24 hrs.
give ursolic acid (I), m. 287-90°, [α]D20 67.0° (c
0.68, iso-PrOH) and 68.4° (c 1.49, dioxane); Me ester, m.
167-8°; Ac derivative, m. 285-8°; **mixed**
anhydride with AcOH, m. 197-8°, resolidifying and decomposing
306-8°; Bz derivative of I, m. 282-4°; AcCH2CO derivative, m.
221-2°. The powdered plant (2 kg.) and 150 g. Ca(OH)2 macerated
overnight with 500 ml. H2O and 6 l. MeOH, refluxed 6 hrs., extracted with
boiling MeOH, the solvent removed from the exts., the residue boiled 5
min. with 2N HCl, the mixture made acid to Congo red, cooled, kept 24 hrs.
at 0°, the aqueous solution concentrated, extracted with CHCl3, and the
extract treated
with NaOH gives vincamine, m. 230-1°, λ 228 and 276 mμ,
ε 29160 and 8213; picrate, m. 228-9°. The mother liquors,
after the separation of I, concentrated, most of the tannins and sugars
removed, the
resulting solid chromatographed on cellulose with 4:1:5 BuOH-EtOH-H2O, and

a pale pink band eluted from the column gave 0.3% of a hygroscopic sirup (II). II hydrolyzed with 2.5N H_2SO_4, extracted with EtOAc, and the extract evaporated gave 2,3-(HO)2C6H3CO2H (III), m. 206-7° [2,3-(MeO)2C6H3CO2Me, m. 47°; Me ester (IV) of III, m. 80-1°]; the aqueous liquid neutralized with $BaCO_3$ and chromatographed yielded **glucose** (phenyl-D-**glucosazone**, decompose 204-5°). Methylating o-vanillin with MeI-K_2CO_3, gives 2,3-(MeO)2C6H3CHO which, oxidized and treated with HBr, yields III (p-chlorobenzamidinium salt, decompose 238-9°; 3-$PhCH_2$ ether, m. 212-13°). II with Me_2SO_4 and NaOH forms 2,3-(MeO)2C6H3CO2H, m. 122-3°; the product obtained with CH_2N_2 gives no $FeCl_3$ reaction, and, when methylated 5 times with MeI and Ag_2O and hydrolyzed with 5N H_2SO_4 yields 3,2-HO(MeO)C6H3CO2H (V), m. 151-2°. [Throughout the following, R = β-D- **glucosyl**, R' = tetraacetyl-β-D- **glucosyl**, and R'' = p-MeC6H4SO2.] **Acetylation** of II and treatment with CH_2N_2 gives 2,3-AcO(R'O)C6H3CO2Me (VI), m. 165-6°. III with HCl in MeOH yields IV, which with α-aceto-D-bromoglucose (VIa) gives VI, [α]D20 -28.3° (c 2.02, Me_2CO). III with p-MeC6H4SO2Cl and NaOH gives 2,3-HO(R''O)C6H3CO2H (VII), m. 176-8° (Me ester, m. 87-8°). Similarly, IV gives 2,3-(R''O)2C6H3CO2Me (VIII), m. 133°, and VII. VII with MeI and K_2O_3 yields 2,3-MeO(R''O)C6H3CO2Me (IX), m. 77-8°, hydrolyzed with NaOH to the acid and V. V with MeI and K_2CO_3 gives IV, which yields III with HBr. V with HCl-MeOH gives 3,2-HO(MeO) C6H3CO2Me (X), b12-13 146-8°, nD20 1.5337. X with VIa, like IV above, gives 2,3-MeO(R'O)C6H3CO2Me (XI), m. 119-20°, [α]D20 -35.0° (c 2, Me_2CO). XI with HCl in MeOH (pH 4.5) and Na gives 2,3-MeO(RO)C6H3CO2Me, m. 187-9°, [α]D20 -53.8° (c 1.01, MeOH). The following compds. were also prepared: 2,3-EtO(R''O)C6H3CO2Et, m. 64-5°; 2,3-EtO(HO)C6H3CO2H, m. 130-1° [p-ClC6H4C(:NH)NH2 salt, decompose 217°]; 2,3-AcO(R''O)C6H3CO2H, m. 149°; 2,3-HO(MeSO3)C6H3CO2H, m. 158-9° (Me ester, m. 110-11°), 2,3-MeO(MeSO3)C6H3CO2H, m. 126-7°, 2,3-EtO(MeSO3)C6H3CO2Et, b0.1 142°, nD20.5 1.5066 (acid, m. 116-17°), 2,3-HO(AcO)C6H3CO2 H, m. 137° (monohydrate, m. 85°; Me ester, m. 70-1°, b0.1 102-4°, nD20.5 1.5243), 2,3-MeO(AcO)C6H3CO2Me, b13-14 162-4°, 2,4-HO(AcO)C6H3CO2Me, m. 51-2°; 2-R'O analog, 2,4-HO(R''O)C6H3CO2H, m. 184-5°, 2,4-MeO(R''O)C6H3CO2Me, m. 56-7° (acid, m. 133-4°), 2,4-MeO(HO)C6H3CO2H, decompose 188-9°, 2,4-HO(R''O)C6H3CO2Me, m. 88-9°, 2,4-HO(MeSO3)C6H3CO2H, m. 175° (Me ester, m. 96-7°), 2,4-MeO(MeSO3) C6H3CO2Me, b0.1 158°, nD20 1.5365 (acid, m. 169°); 2,4-R'O(MeSO3)C6H3CO2Me, m. 157°, [α]D19 -31.3° (c 2.2, Me_2CO); 3,4-MeO(R'O)C6H3CO2Me, m. 141°. 3,4-MeO(HO)C6H3CO2H, m. 208-9°, 3,4-EtO(R'O)C6H3CHO, m. 111-13°, [α]D21 -53.2° (2,4-dinitrophenylhydrazone, m. 202-3°); 3,4-MeO(R'O)C6H3CHO, m. 131-8°, [α]D20 -3.5° (c 4.5, Me_2CO).

L11 ANSWER 17 OF 21 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1941:22451 CAPLUS
DOCUMENT NUMBER: 35:22451
ORIGINAL REFERENCE NO.: 35:3560c-i,3561a
TITLE: Color reactions of some aliphatic acids
AUTHOR(S): Roeder, Geo.
SOURCE: Journal of the American Pharmaceutical Association (1912-1977) (1941), 30, 74-6
CODEN: JPHAA3; ISSN: 0003-0465
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB Alkali citrates (but not citric acid) heated in Ac_2O, give deep-red solns. With AcOH instead of Ac_2O, no color was obtained. The test probably involves the formation of alkali acetate which acts as a condensation agent for the formation of a **mixed anhydride** of

acetylated citric and acetic acid; this theory is supported by the fact that citric acid in Ac2O gives the reaction on addition of alkali acetate. Similar color reactions are obtained on adding numerous basic compds. to solns. of a large number of polyearboxylie acids in Ac2O. The following reactions were noted, the tests being made in each case with 50 mg. of acid in 10 cc. of 95% Ac2O by adding a few grains of solids or about 3 drops of liquid bases. Malonic acid: The solution in Ac2O when carefully heated turns yellow, then orange with green fluorescence; on add. of NaOAc, C5H5N, Et3N or C6H5NMe2 and with further heating the color changes to ruby-red with increased fluorescence. Ethylmalonic acid gives no color under the same conditions. Aconitic acid: The hot solution in Ac2O shows first an amethyst and later a green coloration; the addition of KOAc, C5H5N, C6H5NMe2, strychnine or Et3N changes the color to deep purple. A deep red color is produced on adding to the hot solution of the acid in Ac2O a few grains of alkali acetates, alkali salts of other carboxylic acids, trialkylamines, C5H5N, quinoline, strychnine, nicotine, cinchonine, diethylaminoethanol, triethanolamine, etc.; when the acid is dissolved in 1:1 AcOH-Ac2O, a transparent red solution with a bluish shine is obtained after adding the bases. **Acetyl** citric anhydride reacts in the same way. Alkali citrates give the color when heated with Ac2O alone. Agaricin (**acetyl** citric acid):The solution in boiling Ac2O gave the following colors: with alkali acetates and Et3N, ponceau. red with green fluorescence; with C5H5N, light amethyst; with nicotine or strychnine, deep purple; with C6H5NMe2, yellow or yellowish green; the intensity of the colors produced by alkali acetates increases progressively from Li to Cs. Com. samples of different origins were found to vary in purity, and some gave colors without addition of bases. Tartaric. acid: Boiling the solution in Ac2O with alkali acetates, Et3N, quinoline, nicotine, strychnine, C6H5NMe2 or C5H5N gives successively a yellow, orange and finally brown-red; increasing the quantity of C5H5N changes the color to deep green. Acetonedicarbonic acid: The solution in Ac2O gives successively yellow, orange, red and finally brown when alkali acetates or organic bases are added. Ascorbic acid and d-isoascorbic acid: Boiling the solution in Ac2O with NaOAc, KOAc, Et3N or nicotine gives a deep red color with a bluish shine and slight fluorescence; with C5H5N the solution remains colorless; dissolving the vitamin in 1-2 cc. C5H5N, adding Ac2O and boiling gives an orange color which deepens with prolonged heating into reddish brown. **Glucono**-d-lactone: Addition of KOAc, Cs2CO3, nicotine or Et3N to the boiling solution in Ac2O gives a bright brown color; when the quantity of the base is increased and heating continued, a deep brown is obtained; the reaction is much less intense with NaOAc and nearly zero with LiOAc. **Glucokeptonic** laclone: Because of its insoly, in Ac2O the lactone is first covered with some C5H5N and after warming slightly Ac2O is added; the solution remains colorless on boiling; addition of Cs2CO3 with continued boiling produces a wine-red color gradually darkening into reddish brown and dark brown. Hydroxydimethylbutyrolactone gives no visible reaction when boiled with Ac2O and basic compds.

L11 ANSWER 18 OF 21 MEDLINE on STN

ACCESSION NUMBER:	96294011 MEDLINE
DOCUMENT NUMBER:	PubMed ID: 8731216
TITLE:	Conjugates of insulin with copolymers of N-(2-hydroxypropyl) methacrylamide: effects on smooth muscle cell proliferation.
AUTHOR:	Chytry V; Letourneur D; Baudys M; Jozefonvicz J
CORPORATE SOURCE:	Institute of Macromolecular Chemistry, Academy of Sciences of the Czech Republic Prague, Czech Republic.
SOURCE:	Journal of biomedical materials research, (1996 Jun) Vol. 31, No. 2, pp. 265-72. Journal code: 0112726. ISSN: 0021-9304.
PUB. COUNTRY:	United States
DOCUMENT TYPE:	Journal; Article; (JOURNAL ARTICLE)
LANGUAGE:	English
FILE SEGMENT:	Priority Journals

ENTRY MONTH: 199612
ENTRY DATE: Entered STN: 28 Jan 1997
Last Updated on STN: 28 Jan 1997
Entered Medline: 2 Dec 1996

AB The hypothesis that an elevated plasma insulin level contributes to an increase in coronary heart disease has led to studies of the mitogenic effect of native insulin and its conjugates on smooth muscle cells (SMC). In this study, insulin was covalently attached to two water-soluble polymers containing N-(2-hydroxypropyl)methacrylamide using the **mixed anhydride** method. The first polymer was a copolymer of N-(2-hydroxypropyl)methacrylamide and N-methacryloyldiglycine. The second one was a terpolymer of two of the above-given monomers and R-(-)-1-methyl-2-methacryloylamidoethyl 2-acetamido-2-deoxy-beta-D-**glucopyranoside**. Insulin conjugates were isolated and characterized, and the mitogenic effect on SMC was investigated. The results showed that only conjugates of insulin and terpolymers bearing pendant N-**acetyl-glucosamine** groups do not have a mitogenic effect on SMC while maintaining the hypoglycemic activity of insulin. This finding suggests that some inter- or intramolecular interactions of coupled insulin with the sugar moiety(ies) attached to the polymer backbone contribute to the observed effects.

L11 ANSWER 19 OF 21 MEDLINE on STN
ACCESSION NUMBER: 91210013 MEDLINE
DOCUMENT NUMBER: PubMed ID: 2019473
TITLE: Synthesis of glycosylated tuftsins and tuftsin-containing IgG fragment undecapeptide.
AUTHOR: Biondi L; Filira F; Gobbo M; Scolaro B; Rocchi R
CORPORATE SOURCE: Department of Organic Chemistry, University of Padova, Italy.
SOURCE: International journal of peptide and protein research, (1991 Feb) Vol. 37, No. 2, pp. 112-21.
Journal code: 0330420. ISSN: 0367-8377.
PUB. COUNTRY: Denmark
DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)
LANGUAGE: English
FILE SEGMENT: Priority Journals
ENTRY MONTH: 199105
ENTRY DATE: Entered STN: 16 Jun 1991
Last Updated on STN: 16 Jun 1991
Entered Medline: 29 May 1991

AB Syntheses are described of two new tuftsin derivatives containing a 2-acetamido-2-deoxy-D-galactopyranosyl unit alpha- or beta-glycosidically linked to the threonine's hydroxy side chain function and of the glycosylated undecapeptide corresponding to the tuftsin region of the heavy chain of IgG (amino acid sequence 289-299). The glycosylated tuftsins were synthesized by the solution procedure. Fmoc-[Gal NAc(Ac)3 alpha]Thr-OH and Fmoc-[GalNAc(Ac)3 beta]Thr-OH were allowed to react with H-Lys(Z)-Pro-Arg(NO2)-OBzl by the **mixed anhydride** procedure and the resulting glycosylated tetrapeptides were fully deblocked by catalytic hydrogenation followed by treatment with potassium cyanide, purified by ion exchange chromatography and characterized by analytical HPLC, elemental and amino acid analyses, optical rotation, and proton NMR spectroscopy. Synthesis of the glycosylated undecapeptide was achieved by the continuous flow solid phase procedure on 4-hydroxymethylphenoxyacetyl-norleucyl derivatized Kieselguhr-supported resin. Fmoc-amino acid symmetrical anhydrides or pentafluorophenyl esters, in the presence of N-hydroxybenzotriazole, were used as the acylating agents. To mimic the native sequence of the tuftsin region at the Fc-domain of immunoglobulin G a 2-acetamido-2-deoxy-beta-D-**glucopyranosyl** unit was N-glycosidically linked to the amide side chain of Asn 297. The glycosylated asparagine residue was introduced as N2-fluorenylmethyloxycarbonyl-N4-(2-acetamido-3,4,6-tri-O-**acetyl**

-2 -deoxy-beta-D - **glucopyranosyl**)-asparagine pentafluorophenyl ester. After cleavage from the resin the glycopeptide was deprotected, purified by ion exchange chromatography, and characterized by analytical HPLC, amino acid analysis, high voltage electrophoresis, and proton NMR. The conformational features of the glyco-undecapeptide were determined by circular dichroism measurements both in water and in 98% trifluoroethanol. Results of biological assays will be published elsewhere.

L11 ANSWER 20 OF 21 MEDLINE on STN
ACCESSION NUMBER: 89249066 MEDLINE
DOCUMENT NUMBER: PubMed ID: 2720705
TITLE: Synthesis and reactions of O-**acetylated** benzyl alpha-glycosides of 6-O-(2-acetamido-2-deoxy-beta-D-glucopyranosyl)-N-**acetylmuramoyl**-L-alanyl-D-isoglutamine esters: the base-catalysed isoglutamine in equilibrium glutamine rearrangement in peptidoglycan-related structures.
AUTHOR: Keglevic D; Derome A E
CORPORATE SOURCE: Tracer Laboratory, Department of Organic Chemistry and Biochemistry, Ruder Boskovic Institute, Zagreb, Yugoslavia.
SOURCE: Carbohydrate research, (1989 Feb 15) Vol. 186, No. 1, pp. 63-75.
Journal code: 0043535. ISSN: 0008-6215.
PUB. COUNTRY: Netherlands
DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)
LANGUAGE: English
FILE SEGMENT: Priority Journals
ENTRY MONTH: 198907
ENTRY DATE: Entered STN: 6 Mar 1990
Last Updated on STN: 6 Feb 1998
Entered Medline: 5 Jul 1989

AB Condensation of benzyl 2-acetamido-6-O-(2-acetamido-3,4,6-tri-O-**acetyl**-2- deoxy-3-O-[(R)-1-carboxyethyl]-alpha-D-**glucopyranoside** (2) and its 4-acetate (4) with L-alanyl-D-isoglutamine benzyl ester via the **mixed anhydride** method yielded N-(2-O-[benzyl 2-acetamido-6-O-(2-acetamido-3,4,6-tri-O-**acetyl**-2-deoxy-beta-D-**glucopyranosyl**)-2,3-dideoxy-alpha-D-**glucopyranosid** -3-yl]-(R)-lacto yl)-L- alanyl-D-isoglutamine benzyl ester (5) and its 4-acetate (6), respectively. Condensation by the dicyclohexylcarbodi-imide-N-hydroxysuccinimide method converted 2 into benzyl 2-acetamido-6-O-(2-acetamido-3,4,6-tri-O-**acetyl**- 2-deoxy-beta-D-**glucopyranosyl**)-3-O-[(R)-1-carboxyethyl]-2-deoxy-alpha-D-**glucopyranoside** 1',4-lactone (7). In the presence of activating agents, 7 underwent aminolysis with the dipeptide ester to give 5. Zemplen O-deacetylation of 5 and 6 led to transesterification and alpha----gamma transamidation of the isoglutaminyl residue to give N-(2-O-[benzyl 2-acetamido-6-O-(2- acetamido-2-deoxy-beta-D-**glucopyranosyl**)-2,3-dideoxy-alpha-D-**glucopyr** anosid-3-yl]-(R)-lactoyl)-L-alanyl-D-isoglutamine methyl ester (8) and -glutamine methyl ester (9). Treatment of 6 with MgO-methanol caused deacetylation at the GlcNAc residue to give a mixture of N-(2-O-[benzyl 2-acetamido-6-O-(2-acetamido-2- deoxy-beta-D-**glucopyranosyl**)-4-O-**acetyl**-2,3-dideoxy-alpha-D-**glucopyra** nosid-3-yl]-(R)-lactoyl)-L-alanyl-D-isoglutamine methyl ester (11) and -glutamine methyl ester (12). Benzyl or methyl ester-protection of peptidoglycan-related structures is not compatible with any of the reactions requiring alkaline media. Condensation of 2 with L-alanyl-D-isoglutamine tert-butyl ester gave N-(2-O-[benzyl 2-acetamido-6-O-(2-acetamido-3,4,6-tri-O-**acetyl**-2-deoxy-beta-D-**glucopyranosyl**)-2,3-d ideoxy- alpha-D-**glucopyranosid** -3-yl]-(R)-lactoyl-L-alanyl-D-isoglutamine tert-butyl ester (16), deacetylation of which, under Zemplen conditions, proceeded without side-reactions to afford N-(2-O-[benzyl 2-acetamido-6-O-(2-acetamido-2-

deoxy-beta-D- **glucopyranosyl**)-2,3-dideoxy-alpha-D-**glucopyranosid**-3-yl]-(R)-1a cotyl)-L- alanyl-D-isoglutamine tert-butyl ester (17).

L11 ANSWER 21 OF 21 MEDLINE on STN
ACCESSION NUMBER: 77183529 MEDLINE
DOCUMENT NUMBER: PubMed ID: 861980
TITLE: The preparation of carbohydrate-protein conjugates: cyanuric trichloride coupling of 2-aminoethyl glycosides, and mixed-anhydride coupling of 8-carboxyoctyl glycosides to bovine serum albumin.
AUTHOR: King R R; Cooper F P; Bishop C T
SOURCE: Carbohydrate research, (1977 May) Vol. 55, pp. 83-93. Journal code: 0043535. ISSN: 0008-6215.
PUB. COUNTRY: Netherlands
DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)
LANGUAGE: English
FILE SEGMENT: Priority Journals
ENTRY MONTH: 197707
ENTRY DATE: Entered STN: 14 Mar 1990
Last Updated on STN: 14 Mar 1990
Entered Medline: 29 Jul 1977

AB Preparation of the following glycosides is described: 2-aminoethyl beta-D-glycosides of (A) 2-acetamido-3,4,6-trio-O-**acetyl**-2-deoxy-D-**glucopyranose**, (B) 2-acetamido-4-O-(2-acetamido-3,4,6-trio-O-**acetyl**-2-deoxy-beta-D-**glucopyranosyl**)-3,6-di-O-**acetyl**-2-deoxy-beta-D-**glucopyranose** (N,N'-diacetylchitobiose pentaacetate), (C) 4-O-(2,3,4,6-tetra-O-**acetyl**-beta-D-**glucopyranosyl**)-2,3,6-trio-O-**acetyl**-beta-D-**glucopyranose** (cellobiose heptaacetate); 8-carboxyoctyl glycosides of (D) cellobiose, and (E) N,N'-diacetylchitobiose. Conjugates were prepared from (A), (B), and (C) by coupling to bovine serum albumin by cyanuric trichloride and subsequent deacetylation; (D) and (E) were coupled to bovine serum albumin by the **mixed-anhydride** reaction. Conjugates (A) and (B) were insoluble; conjugates (C), (D), and (E) functioned as artificial antigens and gave rise to precipitating antibodies in rabbits. Specificities of the antisera were determined by inhibition studies.

L13 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1977:30080 CAPLUS
DOCUMENT NUMBER: 86:30080
TITLE: Glutathione complexes with mono- or oligosaccharides or sugar alcohols
INVENTOR(S): Mizutani, Akihiro
PATENT ASSIGNEE(S): Meito Sangyo Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 9 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 51100025	A2	19760903	JP 1975-24971	19750303
PRIORITY APPLN. INFO.:			JP 1975-24971 A	19750303

AB Mono- or oligosaccharides or sugar alcs. were activated with cyanogen halides, organic cyanate esters, or **haloformates** and treated with reduced glutathione to give glutathione complexes, useful as thiol stabilizers. The complexes are more resistant to oxidation than glutathione and stabilize other thiols, e.g., cysteamine or penicillamine. Thus, 10 g sucrose in aqueous NaHCO3 was activated with BrCN at ≤5° for 12 min, poured into Me2CO, and the solid stirred with 1 g glutathione in 0.2M phosphate buffer (pH 6.5) at room temperature for 7 h. The mixture was poured into Me2CO and the solid dissolved in M AcOH and fractionated with Sephadex G-25 to give 6.2 g complex containing 15% glutathione. Similar glutathione complexes were prepared with maltose, mannitol, and **glucose**.

L14 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1976:169742 CAPLUS
DOCUMENT NUMBER: 84:169742
TITLE: Lactam compounds and conjugates
INVENTOR(S): Bolz, Gunner; Leute, Richard K.; Soffer, Michael J.; Singh, Prithipal
PATENT ASSIGNEE(S): Syva Co., USA
SOURCE: Ger. Offen., 59 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2521523	A1	19760115	DE 1975-2521523	19750514
FR 2276317	A1	19760123	FR 1975-12665	19750423
FR 2276317	B1	19790427		
CA 1037475	A1	19780829	CA 1975-225944	19750430
JP 51001632	A2	19760108	JP 1975-61424	19750522
JP 63008427	B4	19880223		

PRIORITY APPLN. INFO.: US 1974-484026 A 19740628

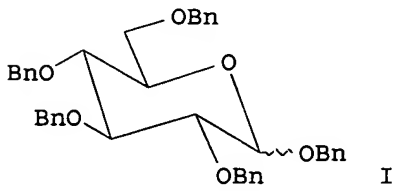
AB Modified drug compds. were prepared which had a lactam function and were disubstituted on the C atom alpha to the carbonyl group. These compds. (hapten acids) were then conjugated to proteins, enzymes, or free radical mols. for use in immunoanal. For example, an aqueous suspension of 10.0 g Na diphenylhydantoin [630-93-3] was treated with an aqueous solution of 10.0 g chloroacetic acid and 10.0 g NaHCO3 to give 2.0 g N3-(carboxymethyl)diphenylhydantoin (I) [741-28-6]. A solution of 124 mg I and 40 μl Et3N in 4 ml anhydrous DMF was treated with 72 μl carbityl **chloroformate** at 0°, and the reaction mixture was added to 160 mg bovine serum albumin in 20 ml H2O to give a conjugate with a hapten number of 51. **Mixed anhydrides** of hapten carboxylic acids with carbityl carbonate were prepared by treating a solution of 0.055 mmole hapten acid and 6.95 μl Et3N in 0.5 ml DMF with 8.5 μl carbityl **chloroformate**. The **mixed anhydrides** were then conjugated with **glucose** 6-phosphate dehydrogenase [9001-40-5]. Hapten acid carbityloxycarbonyl esters and their **glucose** 6-phosphate dehydrogenase complexes, and hapten complexes with 2,2,5,5-tetramethyl-3-amino-1-oxylpyrrolidine were also prepared

L12 ANSWER 1 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2006:358279 CAPLUS
Correction of: 2005:481371
Correction of: 143:26021
TITLE: Acyclic and cyclic carbonic acids and esters, and their sulfur, selenium, and tellurium analogues
AUTHOR(S): Jung, K. W.; Nagle, A. S.
CORPORATE SOURCE: Dept. of Chemistry, University of South Florida, Tampa, FL, 33620-5250, USA
SOURCE: Science of Synthesis (2005), 18, 379-450
CODEN: SSCYJ9
PUBLISHER: Georg Thieme Verlag
DOCUMENT TYPE: Journal; General Review
LANGUAGE: English
AB A review of the preparation and synthetic applications of acyclic and cyclic carbonic acids and esters, and their sulfur, selenium, and tellurium analogs.

L12 ANSWER 2 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1994:558094 CAPLUS
DOCUMENT NUMBER: 121:158094
TITLE: Stereoselective glycosidation
INVENTOR(S): Ishido, Ryoji; Takai, Izumi; Shibazaki, Yoshuki
PATENT ASSIGNEE(S): Tokyo Yatsuka Daigaku, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 39 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 06009674	A2	19940118	JP 1992-191369	19920626

PRIORITY APPLN. INFO.: JP 1992-191369 19920626
OTHER SOURCE(S): CASREACT 121:158094
GI



I

AB The title glycosidation is carried out with hexopyranose 1-carbonic acid ester derivs. and hydroxy compds. in Et2O in the presence of an acid catalyst; the reaction is carried out at a temperature lower or higher than 0°. E.g., 2,3,4,6-tetra-O-benzyl-1-O-(phenoxycarbonyl)-D-glucopyranose (preparation given) was reacted with benzyl alc. in CH2Cl2 at room temperature in the presence of mol. sieve 4A and catalysts CuCl and AgClO4 for 60 h to give 73% a 40:60 mixture of α- and β-I (Bn = benzyl). Many other catalysts, e.g., SnCl2, SnCl4, and solvents, e.g., MeCN, toluene, were also used and their stereoselectivity shown.

L12 ANSWER 3 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1989:490863 CAPLUS
DOCUMENT NUMBER: 111:90863
TITLE: Further evidence for a two-step model of

glucose-transport regulation. Inositol phosphate-oligosaccharides regulate glucose-carrier activity

AUTHOR(S): Obermaier-Kusser, Bert; Muehlbacher, Christa; Mushack, Joanna; Seffer, Eva; Ermel, Britta; Machicao, Fausto; Schmidt, Felix; Haering, Hans Ulrich

CORPORATE SOURCE: Inst. Diabetesforsch., Munich, 8000, Fed. Rep. Ger.

SOURCE: Biochemical Journal (1989), 261(3), 699-705
CODEN: BIJOAK; ISSN: 0306-3275

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The insulin effect on **glucose** uptake is not sufficiently explained by a simple **glucose**-carrier translocation model. Recent studies rather suggest a two-step model of carrier translocation and carrier activation. Several pharmacol. tools were used to characterize the proposed model further. Inositol phosphate (IP)-oligosaccharides isolated from the drug Actovegin, as well as the alkaloid vinblastine, showed a partial insulin-like effect on **glucose**-transport activity of rat fat cells (3-O-methylglucose uptake, expressed as percentage of equilibrium value per 4 s: basal 5.8%, insulin 59%, IP-oligosaccharides 30%, vinblastine 29%) without inducing carrier translocation. On the other hand, two newly developed anti-diabetic compds. (α-activated **carbonic acids**, BM 130795 and BM 13907) induced carrier translocation to the same extent as insulin and phorbol **esters** [cytochalasin-B-binding sites in plasma membranes: basal 5 pmol/mg of protein, insulin 13 pmol/mg of protein, TPA (12-O-tetradecanoylphorbol 13-acetate) 11.8 pmol/mg of protein, BM 130795 10.8 pmol/mg of protein], but produced only 40-50% of the insulin effect on **glucose**-transport activity (basal 5.8%, insulin 59%, TPA 23%, BM 130795 35%). Almost the full insulin effect was mimicked by a combination of phorbol **esters** and IP-oligosaccharides (basal 7%, insulin 50%, IP-oligosaccharides 30%, TPA 23%, IP-oigosaccharides + TPA 45%). None of these substances stimulated insulin-receptor kinase in vitro or in vivo, suggesting a post-kinase site of action. The data confirm the following aspects of the proposed model: (1) carrier translocation and carrier activation are two independently regulated processes; (2) the full insulin effect is mimicked only by a simultaneous stimulation of carrier translocation and intrinsic carrier activity, suggesting that insulin acts through a synergism of both mechanisms; (3) IP-oligosaccharides might be involved in the transmission of a stimulatory signal on carrier activity.

L12 ANSWER 4 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1989:192526 CAPLUS

DOCUMENT NUMBER: 110:192526

TITLE: Preparation of 4-acyloxy-3-hydroxyethylazetidinones as antibiotic intermediates

INVENTOR(S): Schneider, Peter; Ramos, Gerardo; Bersier, Jacques

PATENT ASSIGNEE(S): Ciba-Geigy A.-G., Switz.

SOURCE: Eur. Pat. Appl., 20 pp.
CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 290385	A1	19881109	EP 1988-810264	19880426
EP 290385	B1	19921021		
R: BE, CH, DE, ES, FR, GB, IT, LI, NL, SE				
ES 2045180	T3	19940116	ES 1988-810264	19880426
US 4927507	A	19900522	US 1988-186547	19880427
JP 63297360	A2	19881205	JP 1988-107766	19880502

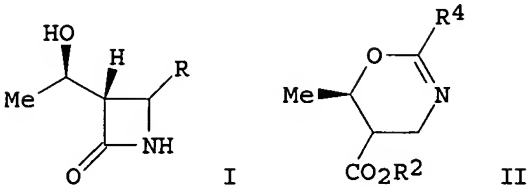
JP 2520447	B2	19960731		
CA 1335971	A1	19950620	CA 1988-565639	19880502
JP 09104678	A2	19970422	JP 1996-13813	19880502
DK 8802389	A	19881105	DK 1988-2389	19880503
DK 170856	B1	19960212		
US 5064761	A	19911112	US 1990-491145	19900309
US 5274188	A	19931228	US 1991-740685	19910806
US 5386029	A	19950131	US 1993-92736	19930716
US 5463047	A	19951031	US 1994-306863	19940915
CA 1337694	A1	19951205	CA 1994-616919	19940919
CA 1339708	A1	19980310	CA 1994-616918	19940919
DK 9500027	A	19950110	DK 1995-27	19950110
DK 171985	B1	19970908		

PRIORITY APPLN. INFO.:

CH 1987-1683	A 19870504
US 1988-186547	A3 19880427
CA 1988-565639	A3 19880502
JP 1988-107766	A3 19880502
US 1990-491145	A3 19900309
US 1991-740685	A3 19910806
US 1993-92736	A3 19930716

OTHER SOURCE(S): MARPAT 110:192526

GI



AB The title compds. (I; R = OCOR1; R1 = alkyl, aryl) were prepared by enantioselective reduction of MeCOCH(CH2NHR3)CO2R2 (R2 = H, **ester** residue; R3 = H, carboxyl or **carbonic acid** half-**ester**-derived acyl), cyclization. of the product to oxazincarboxylate II (R4 = residue of R3), enantiomeric conversion thereof, hydrolysis to (2S,3R)-MeCH(OH)CH(CH2NHR3)CO2R2 [(2S,3R)-III], cyclization. of the latter to I (R = H), and electrochem. oxidation-**esterification** to I (R = OCOR1). MeCOCH(CH2NHBz)CO2Et was shaken 144 h at 33° with baker's yeast in phosphate buffer containing **glucose** to give (2R,3S)- and (2S,3S)-II (R2 = Et, R3 = Bz) which were separated stirred 3 h with SOCl2 in CH2Cl2 to give (5S,6R)- and (5R,6R)-II (R2 = Et, R4 = Ph), the latter of which was isomerized with DBU to (5S,6R)-II (R2 = Et, R4 = Ph). The latter was refluxed 5 h in aqueous HCl to give (2S,3R)-III (R2 = R3 = H) which was heated 6 h at 70° with Ph3P, MnO2, and 2-mercaptopyridine in MeCN to give I (R = H). The latter was electrolyzed 4 h at 50 mA/cm2 in aqueous HOAc containing Bu4N+ BF4- to give I (R = OAc).

L12 ANSWER 5 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:	1987:532664 CAPLUS
DOCUMENT NUMBER:	107:132664
TITLE:	Manufacture of optically active cyanohydrin derivatives
INVENTOR(S):	Ota, Hiromichi; Dobashi, Genichi
PATENT ASSIGNEE(S):	Nissan Chemical Industries, Ltd., Japan
SOURCE:	Jpn. Kokai Tokkyo Koho, 7 pp. CODEN: JKXXAF
DOCUMENT TYPE:	Patent
LANGUAGE:	Japanese

FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 62025997	A2	19870203	JP 1985-164406	19850725

PRIORITY APPLN. INFO.: JP 1985-164406 19850725

AB **Carbonic acid esters** R1CH(CH)OCOR2 (R1, R2 = alkyl, aryl, alkenyl) are contacted with Candida, Penicillium, Aspergillus, Pseudomonas, Arthrobacter, Corynebacterium, or Pichia or their enzyme prepns. to produce optically active cyanohydrins R1CH(CN)OH (R1 = alkyl, aryl, alkenyl) and optically active **carbonic acid esters**. The optically active cyanohydrins are useful as intermediates for organic synthesis . Thus, C. equi was shake-cultured in a medium containing **glucose** 10, polypeptone 7, yeast extract 5, and K2HPO4 5 g at 30° for 2 days, and to this was added 1.1 g mandelonitrile acetate. After cultivation for 3 addnl. days, the culture was extracted to obtain 242 mg mandelonitrile (optical purity >96%).

L12 ANSWER 6 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1978:65992 CAPLUS
DOCUMENT NUMBER: 88:65992
TITLE: Therapeutic agents containing disaccharide derivatives
PATENT ASSIGNEE(S): Meito Sangyo Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 27 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 52048626	A2	19770418	JP 1976-383	19760101
US 4080442	A	19780321	US 1975-623049	19751016

PRIORITY APPLN. INFO.: US 1975-623049 A 19751016

AB Pharmaceuticals containing [OC(Y)Z] [Y = NH or O; Z = -O-(disaccharide residue) or -O-] prepared by treatment of disaccharides with cyanogen halides, organocyanic acid **esters**, or halogenated **carbonic acid** alkyl **esters** are effective in treating liver diseases (acute hepatitis, cholanogiolitic hepatitis, fatty liver, etc.), allergic diseases (allergic coryza, hives, eczema, etc.) and radiation-induced diseases. Thus, 50 g sucrose in 0.045 M NaHCO3 (325 mL) was treated with NCBr (25 g) and the reaction mixture was adjusted to pH 11.0 with 4 N NaOH and maintained at pH 10.9-11.1 and <10° for 80 min by gradual addition of 4N NaOH. The resultant product was poured into 2 L Me2CO to give a white precipitate, which was washed with 500 mL Me2CO, followed by 500 mL MeOH, and freeze-dried to give 35 g powder product (sucrose derivs.). Thus, an injection comprises the sucrose derivs. 3.75, reduced glutathione 0.25, and **glucose** 20 g with addition of distilled H2O to make a final volume of 100 mL.

L12 ANSWER 7 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1969:422272 CAPLUS
DOCUMENT NUMBER: 71:22272
TITLE: Kinetics of formation and decomposition of xanthates of some low molecular weight alcohols and monosaccharides. II. Decomposition experiments and discussion
AUTHOR(S): Philipp, Burkart; Dautzenberg, Horst; Schmiga, Willi
CORPORATE SOURCE: Inst. Faserst.-Forsch., Deut. Akad. Wiss. Berlin, Teltow-Seehof, Fed. Rep. Ger.

SOURCE: Faserforschung und Textiltechnik (1969), 20(4), 179-84
CODEN: FSTXA7; ISSN: 0014-8628
DOCUMENT TYPE: Journal
LANGUAGE: German
AB The rate of decomposition in aqueous solution of xanthates of various alcs. and sugars
was investigated by the polarographic method. In the case of xanthates of monohydroxylic and polyhydroxylic aliphatic alcs., the actual reaction rate constant (kdex) for the dexanthogenation n reaction was proportional to the dissociation constant of the resp. alc. For sugar xanthates, kdex was related as follows: D-glucose < cellobiose < cellulose < Me β-D-glucopyranoside with the stability of the xanthate group at C-2 of aldoses being lowered by etherification of the OH group at C-1. For the C-6 D-glucose xanthate, kdex was 10-50 times higher than kdex for xanthates of monohydroxylic alcs. and two times higher than kdex for MeO(CH2)2OC(S)SK. This indicated that the sugar ring exerts some specific promotional effect on the rate of xanthate decomposition The stability of xanthate residues in cellulose is discussed.

L12 ANSWER 8 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1968:497042 CAPLUS
DOCUMENT NUMBER: 69:97042
TITLE: Selective acylation of D-glucose. Preparation of surface-active D-glucose 6-fatty acid esters
AUTHOR(S): Reinefeld, E.; Korn, H. F.
CORPORATE SOURCE: Tech. Univ. Braunschweig, Brunswick, Fed. Rep. Ger.
SOURCE: Staerke (1968), 20(6), 181-9
CODEN: STRKA6; ISSN: 0038-9056
DOCUMENT TYPE: Journal
LANGUAGE: German
AB D-**Glucose** was partially **esterified** with different acylating reagents (acid chloride, acid imidazolide, mixed anhydride of fatty acid, and Et **ester** of **carbonic acid**) and in different ratios with lauric acid. In any case D-glucopyranose 6-**ester**, D-glucopyranose 1,6-diester and D-glucopyranose 2,6-diester were found as coexisting reaction products. As in the case of p-tolysulfonylation, the primary alc. group is **esterified** most readily, and therefore the formation of 6-**ester** is favored. The ratios of diester isomers, which were isolated by preparative thin-layer chromatog., indicate that after the 6-position, the anomeric hydroxyl and the 2-hydroxyl are acylated most readily. Investigations on D-**glucose** derivs. showed that the course of the partial acylation is not substantially affected when one of these two hydroxyl groups is blocked. In the case of the mixed anhydride, the smallest yield of diesters results, besides the monoester. From D-**glucose** and the fatty acid chlorides, a homologous series of surface active D-glucopyranose 6-**esters** of the natural fatty acids from caproic acid to steric acid were prepared D-Glucopyranose 6-laurate has the greatest surface activity (σ = 29.4 dyne/cm., 0.001M solution).

L12 ANSWER 9 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1963:469366 CAPLUS
DOCUMENT NUMBER: 59:69366
ORIGINAL REFERENCE NO.: 59:12890a-c
TITLE: Borate complexing by five-membered-ring vic-diols. Vapor pressure equilibrium and N.M.R. spectral observations
AUTHOR(S): Mazurek, M.; Perlin, A. S.
CORPORATE SOURCE: Natl. Res. Council Canada, Saskatoon
SOURCE: Canadian Journal of Chemistry (1963), 41(10), 2403-11
CODEN: CJCHAG; ISSN: 0008-4042
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB Thermometric measurement of vapor pressure equilibrium in reaction mixts.

containing borate ion and cis-3,4-dihydroxytetrahydrofuran (I) or D-glucose 5,6-carbonate (II) indicate that complexing involves at least three different equilibrium Borate complexing by I is characterized by a gross change in nuclear magnetic resonance (N.M.R.) spectral characteristics, most striking being a strong, overall, decoupling effect. Alterations in the spectra of II and 5-O-methyl-D-glucose in the presence of borate, when compared with the spectrum of D-glucose, suggest that complex formation by the last sugar proceeds with a pyranose-to-furanose interconversion. The O-C-C-O dihedral angle, within the range 0 to about 40°, does not appear to be a factor determining the stability of borate complexes. Crystalline spirantype complexes of I, II, and of D-threose have been prepared, illustrating the usefulness of borate complexing for the isolation of some furanose sugars and derivs. Solns. of alkali tetraborates are found by thermometric vapor pressure measurements to behave as equimol. mixts. of alkali borate and boric acid.

L12 ANSWER 10 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1963:469365 CAPLUS
DOCUMENT NUMBER: 59:69365
ORIGINAL REFERENCE NO.: 59:12889a-h,12890a
TITLE: Tritium distribution in sugars labeled by the Wilbach method
AUTHOR(S): Simon, H.
CORPORATE SOURCE: Tech. Hochschule, Munich, Germany
SOURCE: Zeitschrift fuer Naturforschung (1963), 18b(5), 360-6
CODEN: ZNTFA2; ISSN: 0372-9516
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB D-Glucose (I), D-fructose (II), D-galactose (III), D-mannose (IV), D-ribose (V), Me tetra-O-acetyl-D-ribofuranoside (VI), sucrose (VII), and D-mannitol (VIII) were exposed to T gas (cf. CA 51, 10359a) under various conditions. The distribution of T in the labeled compds. was determined by measuring the radioactivity of separated (paper chromatography), degraded fragments of HIO4-treated derivs. I, after 1, 6, and 31 days under 200 mm. T, had the following T-distributions in C-1 through C-6: «0.3, «0.3, 2.0; 0, 0, 0.5; 95.0, 92.0, 88.5; 2.8, 2.6, 0.8; 0, 0, 0.6; 4.3, 4.0, 5.8%. Distributions of T in other compds. were as follows: com. I-t, 0.3, 0, 89.4, 4.5, 0, 4.3; com. I-t, 15.1, 0, 42.0, 5.3, 29.7, and 4.0; III, after 9 days under 200 mm. T, 1.4, 0, 53.5, 39.0, 0, 2.4; IV, after 30 days at 3° under 160 mm. T (containing carrier), 2.0, 4.0, 2.0, 85.0, 0, 6.0; V, after 7 days with 200 mc. T, «0.1, «1.0, 95.0, «0.3, 1.4; V from VI, 0.5, 7.6, 0.7, 93.0, 6.3,-; I-t (IX) from VII 0.2, 65.5, 2.9, 18.1, -, 10.1; II-t (X) from VII, 1.2, 0, 5.3, 77.4, -, 10.7%. Irradiation of 400 mg. I upon 2.5 g. activated C with 3 c. of T for 14 days gave a product with 95% of its T at C-2. In I, III, and IV, the distribution between C-4 and C-5 could not be determined and so the sum was listed as C-4. Similarly, after VIII was in 200 mc. T for 21 days, distribution of T in C-1 or 6, C-2 or 5, and C-3 or 4 was 15.8, 53.0, and 30.0%. Specific activities of undild. sugars were: I (after 31 days at 200 mm. T) 1.1 x 107; III 3.2 X 104; IV 7.0 x 105; V 3.6 x 104; VIII 3.0 x 104; and IX 4.3 x 105 counts per min./μmole. pecific activities after dilution with unlabeled sugar were: I, after 1, 6, and 31 days exposure to T, 393.2, 1176, and 3540; II 296.4; III 427.6; IV 1006; V 293; VIII 572; IX 370; X 601 counts per min./μ mole. Labeled aldoses were converted to the glycoside and then to the osazone. Tritium on C-1, C-2, and C-6 of original labeled aldoses was determined by comparison of osazones with the osazone of I which had been labeled at these positions (CA 53, 1160g; 56, 12993g). I-1-t changed little in specific radioactivity («4%) when converted to the corresponding osazone, N-phenylosotriazole, N-phenyltriazolaldehyde, and N-phenyltriazole carboxylic acid. With II, T in C-1 could not be determined directly due to H elimination and a kinetic isotope effect on isotope formation. It was estimated that approx. 2÷3 of T remained at C-1 during osazone formation.

I-2-t (XI) had specific activities of 395.7, 395.6, and 393.4 counts per min./μ mole indicating no measurable isotope effect. After recrystn., the osazone (XII) of XI showed 0.3% of the radioactivity of XI. Thus, the difference in radioactivity between XI and XII was attributed to the activity at C-2. Direct determination of T in C-5 of pentoses or in C-6 of hexoses was difficult due to ease of overoxidn. of CH2O (from C-6) (XIII) with HIO4 resulting in T enrichment of XIII. Upon heating D-arabino-hexose phenylosazone of D-lyxo-hexose phenylosazone in EtOT with alkali, fast T fixation occurs at C-1 and slower fixation occurs at C-3 of the sugar derivative For T content on C-4 and C-5 of hexoses, the 6-Obenzoyl-N-phenylosatriazoles were prepared and split with HIO4, extracted with Et2O, and subjected to thin layer chromatography. Spots recognizable in ultraviolet light as BzCH(OH)CHO (XIV) were extracted with Et2O and XIV was isolated as its 2,4-dinitrophenylhydrazone in 30% yield. HCO2H (XV) (derived from C-4) was isolated from the aqueous phase. Purification of labeled compds. was accomplished most efficiently by descending paper chromatography. Thus, 30-35 mg. of the radioactive portion of T-labeled compds. eluted from thick filter paper with 4:1 PhOHH2O was detected with a windowless counting tube and diluted with the corresponding nonlabelled compound The N-glycoside of the diluted eluate had constant specific activity after 2 recrystns. The method of Weygand, et al. (CA 34, 833) was used to prepare most of the glycosides. N-D-Ribosylaniline was prepared according to Berger, et al. (CA 40, 31014). Osazones (XVI) were prepared in MeOCH2CH2OH and most osotriazoles (XVII) were prepared from XVI by the method of Simon, et al. (CA 56, 12993g). D-lyxo-Hexose phenylosotriazole was prepared by suspending 415 mg. galactosazone in 50 ml. H2O, adding 585 mg. pure CuSO4.5H2O in 2 ml. H2O, heating with stirring in an oil bath at 120-130°, treating with activated C, filtering hot, and removing Cu by stirring 20 min. with 2 ml. Dowex 50-X8 (200-400 mesh), filtering through more of the same resin, washing with H2O, stirring 30 min. with 13 ml. Dowex 2-X8 (20-50 mesh) in OH form, filtering, rewashing with H2O, and evaporating on a H2O bath at 40-50° under vacuum. The residue (280 mg.) was deodorized with C in iso-PrOH and precipitated with Et2O to give 85% yield of D-lyxo-hexose phenylosotriazole, m. 110°. Labeled VIII was degraded with HIO4 by heating 728 mg. in Sorensen buffer (pH 5.4) with 4.7 g. NaIO4 for 40 min. at 60°. After destruction of IO4- and IO1-, precipitation with dimedon gave a 58% yield of product, m. 190°. 1,6-Dibenzoylmannitol (XIX), m. 179° (2 recrystns. from EtOH), was prepared according to Brigl and Gruener (CA 26, 3780). XIX (550 mg.) was stirred with 1.02 g. NaIO4 in 100 ml. H2O on a steam bath, quickly cooled to 0°, and extracted 5 times with 35-ml. portions of Et2O. The residue (430 mg.) from the Et2O layer was refluxed 30 min. with 45 ml. EtOH, 4.3 ml. HOAc, and 570 mg. 2,4-(O2N)2-C6H3NHNH2 to yield 820 mg. crude product.

L12 ANSWER 11 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1963:441953 CAPLUS
DOCUMENT NUMBER: 59:41953
ORIGINAL REFERENCE NO.: 59:7623c
TITLE: Oxidation and transesterification reactions of sugar derivatives in dimethyl sulfoxide
AUTHOR(S): Henseke, G.; Hanisch, G.
CORPORATE SOURCE: Bergakad., Freiberg/Sa., Germany
SOURCE: Angew. Chem. (1963), 75, 420
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB Oxidation of 5,6-anhydro-1,2-O-isopropylidene-α-D-glucose with Me2SO-BF3 gave 1,2-O-isopropylidene-D-gluco-hexodialdose, characterized as the phenylhydrazone, and, after hydrolysis, as the bis(phenylhydrazone), m. 170-2°. Me2SO-KHCO3 did not oxidize 1,2-O-isopropylidene-6-O-p-tolylsulfonyl-α-D-glucose, but converted it into 1,2-O-isopropylidene-α-D-glucose 5,6-carbonate.

L12 ANSWER 12 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1963:403842 CAPLUS
DOCUMENT NUMBER: 59:3842
ORIGINAL REFERENCE NO.: 59:757e-f
TITLE: Synthesis of glycopeptides
AUTHOR(S): Lieflaender, Manfred
CORPORATE SOURCE: Max-Planck Ges., Goettingen, Germany
SOURCE: Naturwissenschaften (1962), 49, 541
CODEN: NATWAY; ISSN: 0028-1042
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB (Z = carbobenzoxy group, DMF = HCONMe2, benz = benzyl ester.) N-aminoacyl-D-glucosamine- or D-glucosamine-containing peptides were prepared from the corresponding N-carbobenzoxy-L-amino acids (I) or N-carbobenzoxy-L-peptides (II) and D-glucosamine using ClCO2Et as a coupling reagent (N-aminoacyl groups, m.p., [α]25D, and % yield product given): N-Z-Gly, 183°, 43.2° (c 2.2, MeOH), 58; N-Z-L-Leu, 188°, 19.7° (c 2.2, MeOH), 69; Nα.ε-Z2-L-Lys, 137°, 22.6° (c 1.9, MeOH), 81; N,O-Z2-L-Tyr, 196°, 22.4° (c 2.0, MeOH), 61; N-Z-L-Leu-L-Leu, 202°, -4.9° (c 2.0, MeOH), 67; N-(NεNα-Z2-L-Lys-Gly, 182°, 42.2° (c 0.7, DMF), 54; N-(Z-L-Glu-α-benz, 153°, -5.2° (c 1.9, DMF), 56; N-Z-L-Glu-γ-benz, 194°, 55.2° (c 1.3, DMF), 62; N-Z-L-Asp-α-benz, 193°, 34.8° (c 2.0, DMF), 66; N-Z-L-Asp-β-benz, 182°, 33.3° (c 2.0, DMF), 65. The compds. gave no reaction with Ehrlich's reagent and gave by catalytic hydrogenolysis over Pd-C in methanolic HCl the hydrochloride of the corresponding free aminoacyl-D-glucosamine or peptide-D-glucosamine in good yields.

L12 ANSWER 13 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1962:477400 CAPLUS
DOCUMENT NUMBER: 57:77400
ORIGINAL REFERENCE NO.: 57:15448g-i,15449a
TITLE: Magnesium glucomonocarboxylate. Toxicology and pharmacology
AUTHOR(S): Benzi, G.
CORPORATE SOURCE: Univ. Pavia, Italy
SOURCE: Farmaco, Edizione Pratica (1962), 17, 280-90
CODEN: FRPPAO; ISSN: 0430-0912
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB Acute toxicity of Mg glucomonocarboxylate(I) was tested in mice, rats, guinea pigs, and rabbits (animal, administration, and L.D.50 in mg/kg. given): mouse intravenous, 630, intraperitoneal, 2830, oral 21,260; rat intraperitoneal, 2780, oral, 18,170; guinea pig, intravenous, 770, intraperitoneal, 4010; rabbit, intravenous, 840. I was administered to rats intraperitoneally (40 mg./kg. for 34 days) and orally (2% of the diet of 30 days) and after this time no abnormal result was observed. Rats, during pregnancy, treated intraperitoneally with 80 mg./kg./day, delivered normally. In anesthetized guinea pigs weighing 315-830 g., intraduodenally treated with 1 cc./kg. 50% I at 37°, a reflex contraction of the gall bladder was demonstrated. Expts. carried out on rabbits weighing 2350-2730 g. and surgically provided with a cholecyst fistula (the quant. variations of biliary secretion were observed before and after the administration of 1 cc./kg. 30% I) showed that the treatment produced an increase in biliary secretion, amounting to 2-16.2%. No variations in the composition of total cholic acids were observed. In vitro expts. by Benzi and Crema technique (Benzi and Crema, Boll. Society Ital. Biol. Sper. 36, 664 (1960)) showed that I inhibited the contracting action of carbamylcholine on the isolated Oddi sphincter of calf.

L12 ANSWER 14 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1960:134619 CAPLUS
DOCUMENT NUMBER: 54:134619
ORIGINAL REFERENCE NO.: 54:25743f-h
TITLE: Oxyalkylated glucoses for demulsifying petroleum emulsions
INVENTOR(S): De Groote, Melvin; Pettingill, Owen H.
PATENT ASSIGNEE(S): Petrolite Corp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2945024		19600712	US 1957-653447	19570417

AB Treatment of glucose (I) with ethylene oxide (II) and butylene oxide (III) results in a cogeneric mixture of a homologous series of glycol ethers of I which are useful as demulsifying agents for H2O-in-oil emulsions. Although I may be oxyalkylated by both II and III simultaneously, it is preferred to oxybutylate first and then to cause this intermediate to react with II to give the final products. Oxybutylation of glucose is carried out in an autoclave at 135-150° at a maximum pressure of 50 lb./sq. in.; III is added to I by a stepwise technique and NaOMe is used as the catalyst. Best results are obtained when III consists of the straight-chain isomers with little or no isobutylene oxide. Oxyethylation is carried out at maximum temps. of 130-5° and pressures of 10-15 lb./sq. in. Ethylene carbonate may be used instead of II without use of pressure. The average composition of the products stated in terms of the initial reactants lies within a 5-sided figure identified by the following points: 10% I, 85% II, and 5% III; 1.5% I, 85.0% II, and 13.5% III; 1.5% I, 40.0% II, and 58.5% III; 20% I, 40% II, and 40% III; 20% I, 70% II, and 10% III. The min. I content is 1.5%. The products have other applications, such as reaction with alkyleneimines to produce cation-active materials, as emulsifying agents, detergents, and solvents.

L12 ANSWER 15 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1955:33928 CAPLUS
DOCUMENT NUMBER: 49:33928
ORIGINAL REFERENCE NO.: 49:6539d-g
TITLE: The amount of stable esters of carbonic acid [present] in wine
AUTHOR(S): Kozenko, E. M.
CORPORATE SOURCE: Inst. Food Ind., Krasnodar
SOURCE: Vinodelie i Vinogradarstvo SSSR (1952), 12(No. 4), 25-8
CODEN: VIVSA6; ISSN: 0042-6318
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB **Carbonic acid esters** (I) play a significant role in the formation of wine flavor. The best known of the **esters** is di-Et carbonate, EtOCOOEt (II) (b. 126°, d. 0.976). The amount of I present in must and different wines was studied by measuring the CO2 evolved on hydrolysis of I. H2SO4 (4N) was found to be the best hydrolyzing agent as far as the hydrolysis of II is concerned. However, H2SO4 is also an oxidizing agent. From the wine constituents tested (sucrose, fructose, **glucose**, arabinose, tartaric, malic, lactic, acetic, and amino acids, MeOH, EtOH, glycerol, and tannins) the wine sugars also evolved CO2 upon treatment with 4N H2SO4. Therefore, I were extracted from the exptl. samples by Et2O (acidified by H2SO4) before the hydrolysis. The accuracy of the CO2 measurement was ±0.1 ml., which corresponds to ±0.4 mg. of II. Sparkling wines (champagne) contain the largest amount of I (61-70 (young) and 90-98 mg./l. (old) (expressed as II)) as compared with common table wines (17-18 mg./l.); in must no I is

present. The amount of I increases during the alc. fermentation of must, reaching a maximum at the end of the fermentation. After removal of CO2 from the wine some of the I are hydrolyzed; however, the hydrolysis takes place very slowly. The presence of a large amount of I (up to 10 g. II/l.) in must does not affect the normal course of the alc. fermentation.

L12 ANSWER 16 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1950:12472 CAPLUS
DOCUMENT NUMBER: 44:12472
ORIGINAL REFERENCE NO.: 44:2452a-i,2453a
TITLE: Desoxy sugars. IV. Synthesis of 2-desoxy-D-ribose from D-erythrose
AUTHOR(S): Overend, W. G.; Stacey, M.; Wiggins, L. F.
CORPORATE SOURCE: Univ. of Birmingham, UK
SOURCE: Journal of the Chemical Society (1949) 1358-63
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 44:12472

AB cf. preceding and following abstrs. Crude brucine erythronate (3 g.), obtained by hydrolyzing oxidized starch, was converted by (CO2H)2 into the lactone, and by means of Ac2O and dry HCl into 2,3-diacetyl-D-erythronolactone, m. 50-1.5°. Ca D-arabonate (I), m. 99-101° (from H2O), [α]D21 -6.8°, was formed from D-arabinose and Br, followed by aeration, treatment with Ag2O, filtration, precipitation with H2S, filtration, and heating with CaCO3. Solns. of 14.89 g. Ba(OAc)2.H2O in 43 ml. H2O and 7.31 g. Fe2(SO4)3 in 43 ml. H2O were added gradually to 1.42 l. H2O and the boiling mixture treated slowly with 123.4 g. I, filtered through carbon, treated with 86.04 ml. (100 volume) H2O2, cooled to 40°, and again treated with the same amount of H2O2. After filtration and evaporation in vacuo, the mixture was treated with an excess of MeOH, filtered, and evaporated, giving a pale yellow sirup setting to noncryst. glassy D-erythrose (II), [α]D14.5 -18.5° (equilibrium value in H2O), converted into 2,3-propylidene-α,β-methyl-D-erythroside (III), b10 100°, [α]D14.5 -55.5° (CHCl3), by shaking in dry Me2CO, MeOH, and 0.2% H2SO4 with CuSO4. (The L-isomer of III, b2 45-50°, [α]D 57.4° (cf. Felton and Freudenberg, C.A. 29, 7288.4) With 0.1 N H2SO4 at room temperature, III gave II; phenylosazone, m. 160-2.5° (from EtOH). Two other methods for preparing II were also carried out. Triacetylglucal (12.95 g.) was heated 15 min. with H2O, concentrated in vacuo, extracted with Et2O, washed, dried, heated with Ac2O and AcONa at 100° 3 hrs., evaporated, and EtOH distilled twice over the residue, which was then reextd. with Et2O and dried, yielding 1,4,6-triacetylpseudoglucal (IV), b0.01 115-25° [α]D18 66.8° (CHCl3), nD19 1.4839, decolorizes Br-H2O. IV (0.5 g.) in Et2O hydrogenated with Pt catalyst gave triacetyldidesoxyglucose, C12H18O7, oil, b0.01, about 120-30° [α]D15 32.63° (CHCl3), nD15 1.4548. Ozonization of IV (0.595 g.) in AcOH until Br in CCl4 was no longer decolorized gave 0.42 g. of the 2,4-di-Ac derivative of II, readily hydrolyzed to II by 0.05 N HCl. 1,2-Isopropylideneglucofuranose 5,6-carbonate (V) (cf. Haworth and Porter, C.A. 24, 1350), m. 226°, [α]D20 -37.4°, was treated in pyridine at 0° with MeSO2Cl, giving the 3-MeSO2 derivative of V, needles, m. 136-7°, [α]D18.5 -22.1°. V heated in EtOH with concentrated HCl at 70-75° formed glucofuranose 5,6-carbonate, m. 179°, [α]D17 18.1° (H2O). Warmed with an excess of aqueous Ba(OH)2 at 70°, V gave 1,2-isopropylideneglucofuranose, m. 158-9°, [α]D16.5 -13.6° (H2O). V (1 g.) at 45° in 25 cc. MeOH containing 0.3 cc. H2SO4 gave, after BaCO3 treatment, Me glucofuranoside 5,6-carbonate, m. 142-4° (from MeOH-Et2O), [α]D22 -64°, which when oxidized in C6H6 with Pb(OAc)4 in AcOH at 0° followed by hydrolysis with Ba(OH)2, gave II. To 5.4 g. II in dry MeOH was added 33.7 cc. MeNO2, followed by 55 cc. MeOH containing 1.42 g.

Na, and, after shaking 24 hrs., the mixture was treated with 40 cc. Et2O, causing the separation of a solid (VI) which, in H2O, was passed through a Zeocarb resin column, and the effluent evaporated The original mother liquors from VI were treated with AcOH, the mixture evaporated, given a treatment similar to that received by VI, and the combined effluents from the resin-bearing column dried and acetylated, followed by distillation of the AcOH and heating with C6H6 and NaHCO3; filtration and evaporation gave 1-nitro-D-erythro-3,4,5-triacetoxy-1-pentene (VII), yellow oil, nD18 1.4553, [α]D20 -8.6° (EtOH), undergoing decomposition on attempted distillation at 0.005 mm. and 160-70°. After 3 months VII crystallized and m. 116-18° (from aqueous EtOH). VII in dry MeOH was hydrogenated with Pd at room temperature, filtered, evaporated to a sirup, treated 1 hr. with N NaOH, the mixture added to aqueous H2SO4 at room temperature, and the solution neutralized with BaCO3, stirred with carbon, filtered, and evaporated, giving a residue which reduced Fehling solution and gave a blue Dische color, and which when mixed with H2O, PhNH2, and enough EtOH to insure a homogeneous solution at 0°, gave, after evaporation, 2-desoxy-D-ribose anilide (VIII), m. 175-6°, [α] 20.5° (equilibrium value in alc.). When hydrolyzed with 0.5% (CO2H)2, VIII gave 2-desoxy-D-ribose, m. 87-90° (from AcOEt, and then from iso-PrOH), [α]D22 -55.2° (H2O) (cf. Levene and Mori, C.A. 24, 2111, who give the m.p. 80°), giving VIII on reanilation.

L12 ANSWER 17 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1947:37415 CAPLUS
DOCUMENT NUMBER: 41:37415
ORIGINAL REFERENCE NO.: 41:7420a-g
TITLE: 2-Methoxy-6-chloro-9-[2-hydroxy-3-(butylethylamino)propylamino]acridine
INVENTOR(S): Shonle, Horace A.; Corse, Joseph W.
PATENT ASSIGNEE(S): Eli Lilly and Co.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2424063		19470715	US 1944-548499	19440807

GI For diagram(s), see printed CA Issue.
AB Antimalarials of this type are prepared by condensation of a diamino alc. with 6,9-dichloro-2-methoxyacridine (I) as in the following examples. Example 1:To 53 g. O.CH2.CMeCH2Cl (II) in 50 cc. 2-methoxy-ethanol (III) is added 36.5 g. MeNHPr in 50 cc. III. After standing overnight, the mixture is stirred with a solution of 92.5 g. K phthalimide (IV) and 5 g. NaI in 100 cc. III 5.5 hrs. at about 130°. The solvent is evaporated and the residue dissolved in dilute HCl. The solution is filtered and aqueous NaOH added. The precipitate of crude N-[2-hydroxy-2-methyl-3-(methylpropylamino)propyl]phthalimide, in 62-g. yield, is dissolved in 250 cc. absolute alc., and 12 g. 85% N2H4.H2O is added. After 4 hrs., dilute HCl is added, the precipitate filtered out, the filtrate evaporated to dryness, and 12.5 N NaOH solution is added to the residue to liberate crude 1-amino-2-methyl-3-(methylpropylamino)-2-propanol; 9 g., stirred at 100° 2 hrs. with 4 g. I and 50 cc. phenol and dilute aqueous NaOH added, liberates 1-(6-chloro-2-methoxy-9-acridylamino)-2-methyl-3-(methylpropylamino)-2-propanol (SN 5559); di-HCl salt m. 160-3°. Example 2:iso-PrNHPr (40.4 g.) is dissolved in 100 cc. III, and 38.6 g. epichlorohydrin (V) is added dropwise with cooling. After standing overnight, the mixture is stirred with a solution of 74 g. IV and 5 g. KI in 75 cc. III 6 hrs. at 130°. The solvent is vacuum-evaporated and the residue dissolved in

dilute HCl. The solution is filtered and made basic, precipitating N-[2-hydroxy-3-(isopropylpropylamino) propyl]phthalimide. With acid hydrolysis this yields 1-amino-3-(isopropylpropylamino)-2-propanol, which is condensed with I to form 1-(6-chloro-2-methoxy-9-acridylamino)-3-(isopropylpropylamino)-2-propanol (SN 5561); the di-HCl salt m. 205-8°. Example 3:V (46.3 g.) is slowly added to 50.5 g. EtNHBu in 100 cc. MeOH and let stand overnight. The MeOH is evaporated and the residue is used in the manner of Example 2 for the successive prepns. of N-[3-(butylethylamino)-2-hydroxypropyl] phthalimide, 1-amino-3-(butylethylamino)-2-propanol, and 1-(butylethylamino)-3-(6-chloro-2-methoxy-9-acridylamino)-2-propanol (SN 5564) (di-HCl salt, m. 199-201°). Example 4:With MeNHPr as a starting material, the following are successively prepared as in Example 2: N-[2-hydroxy-3-(methylpropylamino)propyl]-phthalimide, 1-amino-3-(methylpropylamino)-2-propanol, and 1-(6-chloro-2-methoxy-9-acridylamino)-3-(methylpropylamino)-2-propanol (SN 5557) (di-HCl salt, m. 153-6°). Example 5:EtNH2 and cyclopentanone are hydrogenated together at about 100° and 1500 lb./sq. in. in the presence of Raney Ni. The Ni is removed and the mixture distilled The fraction b29 119-20° is N-ethylcyclopentylamine. It is used in accordance with the previous examples for the successive prepns. of 1-amino-3-(cyclopentylethylamino)-2-methyl-2-propanol and 1-(6-chloro-2-methoxy-9-acridylamino)-3-(cyclopentylethylamino)-2-methyl-2-propanol (the structural formula in the patent appears erroneous).

L12 ANSWER 18 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1942:26798 CAPLUS
DOCUMENT NUMBER: 36:26798
ORIGINAL REFERENCE NO.: 36:4098h-i,4099a-c
TITLE: Synthesis of some new glucose and gentiobiose derivatives
AUTHOR(S): Reynolds, Delbert D.; Kenyon, Wm. O.
SOURCE: Journal of the American Chemical Society (1942), 64, 1110-12
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB β-d-Glucose 1,2,3,4-tetraacetate (I) (R. and Evans, C. A. 33, 143.8) (20 g.) and 5 g. drierite in 50 cc. dry C5H5N, treated slowly with 2.8 g. COCl2 in 10 cc. dry PhMe and then with 50 cc. C6H5N, shaken at room temperature for 15 hrs. and then warmed on a steam bath for 2 hrs., give 82% of bis(1,2,3,4-tetraacetyl-β-d-glucosyl) carbonate (II), m. 198-9°, [α]546126.5 12.15° (CHCl3, cc 4.026). II (20 g.) in 200 cc. 32% HBr in AcOH, allowed to stand 2 hrs. at room temperature and treated with 200 cc. CHCl3, with crystallization from CHCl3-ether, gives 80% of bis(1-bromo-2,3,4-triacetyl-β-d-glucosyl) carbonate (III), m. 147-8°, [α]546120.5 258° (CHCl3, c 4.018). Ag2O (4 g.), 10 g. drierite and 75 cc. absolute MeOH in a flask wrapped in black paper, stirred 1 hr. and treated during 0.5 hr. with 5 g. III in 25 cc. CHCl3 (with stirring overnight), give 87% of bis(2,3,4-triacetyl-β-d-methylglucosidyl) carbonate, m. 191-2°, [α]546126 -75°. Ag2O (10 g.), 20 g. drierite and 9.2 g. I in 65 cc. CHCl3, stirred 1 hr. and treated with 10 g. III in CHCl3 during 1 hr. with stirring for 22 hrs., give 40% of bis(1,2,3,4,2',3',4'-heptaacetyl-β-gentiobiosyl) carbonate (IV), m. 257-8°, [α]546126 -28.8° (CHCl3, c 2.31). Stirring IV and MeONa in CHCl3-MeOH for 1 hr. at room temperature gives 76% of β-d-gentiobiose, characterized as the octaacetate, m. 195-6° (cf. loc. cit.). III is very stable and did not show noticeable decomposition after exposure to laboratory conditions for several months.

L12 ANSWER 19 OF 20 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1939:67042 CAPLUS

DOCUMENT NUMBER: 33:67042
ORIGINAL REFERENCE NO.: 33:9625h-i,9626b-c
TITLE: Defect spots in cellulose molecules
AUTHOR(S): Staudinger, H.; Sohn, A. W.
SOURCE: Naturwissenschaften (1939), 27, 548-9
CODEN: NATWAY; ISSN: 0028-1042
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB Evidence from viscosity of nitrated cellulose compared with cellulose precipitated from Schweizer reagent indicates that the mols. of fiber cellulose contain **ester**-like compds. saponifiable by alkali (C. A. 28, 6999.7). Hence there are 2 types of cellulose chains: the normal one built up of **glucose** groups with an uninterrupted sequence of pyranoid rings; the other one has reactive defect spots which contain **ester** groups, saponified by alkali. The second type can be formed from the former by oxidative splitting off of one **glucose**, thus giving a **carbonic acid ester**. Such a group of general form .O.CHCO2H.CHCH2OH.O.C:O. does not decompose with HNO3. Viscosity detns. in Schweizers reagent give the polymerization degree of the normal cellulose chains; viscosity measurements of native cellulose after nitration give results on polymerization of total chains including **ester** groups. With a polymerization number of 740 prior to alkali treatment the number is reduced to 185 in a given example after solution in Schweizer reagent. The sensitivity of the cellulose with defects to alkali treatment is important for tech. use. Oxidation processes such as bleaching or peroxide treatment can bring about the formation of defects.

L12 ANSWER 20 OF 20 MEDLINE on STN
ACCESSION NUMBER: 90026256 MEDLINE
DOCUMENT NUMBER: PubMed ID: 2803236
TITLE: Further evidence for a two-step model of glucose-transport regulation. Inositol phosphate-oligosaccharides regulate glucose-carrier activity.
AUTHOR: Obermaier-Kusser B; Muhlbacher C; Mushack J; Seffer E; Ermel B; Machicao F; Schmidt F; Haring H U
CORPORATE SOURCE: Institut fur Diabetesforschung, Munchen, Federal Republic of Germany.
SOURCE: The Biochemical journal, (1989 Aug 1) Vol. 261, No. 3, pp. 699-705.
Journal code: 2984726R. ISSN: 0264-6021.
PUB. COUNTRY: ENGLAND: United Kingdom
DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)
LANGUAGE: English
FILE SEGMENT: Priority Journals
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AB The insulin effect on **glucose** uptake is not sufficiently explained by a simple **glucose**-carrier translocation model. Recent studies rather suggest a two-step model of carrier translocation and carrier activation. We used several pharmacological tools to characterize the proposed model further. We found that inositol phosphate (IP)-oligosaccharides isolated from the drug Actovegin, as well as the alkaloid vinblastine, show a partial insulin-like effect on **glucose**-transport activity of fat-cells (3-O-methylglucose uptake, expressed as % of equilibrium value per 4 s: basal 5.8%, insulin 59%, IP-oligosaccharides 30%, vinblastine 29%) without inducing carrier translocation. On the other hand, two newly developed anti-diabetic compounds (alpha-activated **carbonic acids**, BM 130795 and BM 13907) induced carrier translocation to the same extent as insulin and phorbol **esters** [cytochalasin-B-binding sites in plasma membranes: basal 5 pmol/mg of protein, insulin 13 pmol/mg of protein, TPA (12-O-tetradecanoylphorbol 13-acetate) 11.8 pmol/mg of protein, BM 130795

10.8 pmol/mg of protein], but produce also only 40-50% of the insulin effect on **glucose**-transport activity (basal 5.8%, insulin 59%, TPA 23%, BM 130795 35%). Almost the full insulin effect was mimicked by a combination of phorbol **esters** and IP-oligosaccharides (basal 7%, insulin 50%, IP-oligosaccharides 30%, TPA 23%, IP-oligosaccharides + TPA 45%). None of these substances stimulated insulin-receptor kinase in vitro or in vivo, suggesting a post-kinase site of action. The data confirm the following aspects of the proposed model: (1) carrier translocation and carrier activation are two independently regulated processes; (2) the full insulin effect is mimicked only by a simultaneous stimulation of carrier translocation and intrinsic carrier activity, suggesting that insulin acts through a synergism of both mechanisms; (3) IP-oligosaccharides might be involved in the transmission of a stimulatory signal on carrier activity.